EPA Region 5 Records Ctr.
259716

FINAL
MAIN SITE EVALUATION
WORK PLAN

For the
Former Celotex Facility
2800 South Sacramento Avenue
Chicago, Illinois 60623

Prepared for

Honeywell International Inc.

October 2006

Prepared by

CH2MHILL

Executive Summary

This work plan presents the procedures that will be used to evaluate clay, fill, and gravel materials that have been placed during the last 10-12 years on the former Celotex Corporation (Celotex) Site (Main Site) located at 2800 South Sacramento Avenue in Chicago, Illinois. The evaluation findings will be utilized to support decision-making related to the Main Site remedy. The work plan has been prepared on behalf of Honeywell International Inc. (Honeywell).

The purpose of this evaluation is to:

1) Gather physical and analytical data concerning the clay, fill, and gravel materials
2) Survey the existing top of the gravel cap and, using data from the soil borings, determine the thickness of the gravel cap, clay cover, and fill materials between the cap and cover across 22 acres of the Main Site.

Upon completion of the evaluation proposed herein, a Main Site Evaluation Report will be developed to document the findings of the evaluation and will be submitted to the United States Environmental Protection Agency (USEPA).

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Acronyms and Abbreviations

AOC	Administrative Order by Consent
bgs	below ground surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CIC	Community Involvement Coordinator
DQO	data quality objective
EE/CA	*Engineering Evaluation, Cost Analysis*
ERM	Environmental Resources Management Group
HSA	hollow stem auger
HSP	Health and Safety Plan
ISGS	Illinois State Geological Survey
MWRDGC	Metropolitan Water Reclamation District of Greater Chicago
NELAP	National Environmental Laboratory Accreditation Program
PAH	polycyclic aromatic hydrocarbon
PID	Photoionization detector
PCB	Polychlorinated Biphenyl
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
RPM	Remedial Project Manager
SOP	Standard Operating Procedure
SPLP	Synthetic Precipitation Leaching Procedure
SVOC	Semivolatile Organic Compounds
USEPA	U.S. Environmental Protection Agency
USCS	Unified Soil Classification System
USGS	U.S. Geological Survey
VOC	Volatile Organic Compounds

SECTION 1

Introduction

This work plan, prepared on behalf of Honeywell International Inc. (Honeywell), presents proposed Cap, Fill, and Cover (as herein defined) evaluation activities for the Main Site. Evaluation findings will be used to support decision-making related to the Main Site remedy. The location of the Main Site is illustrated on Figures 1-1 and 1-2.

This work plan provides a description of the tasks that will be performed to complete the investigation phase of the cap evaluation. Health and safety requirements and procedures for the work are presented in the Health and Safety Plan (HSP). Detailed descriptions of analysis procedures and quality assurance protocols are presented in the Quality Assurance Project Plan (QAPP) and QAPP Addendum. The QAPP for this project was prepared for the residential sampling program that is underway. It was prepared in accordance with the U.S. Environmental Protection Agency (USEPA), Region 5 guidance and was approved by the USEPA. The QAPP Addendum provides project specific information for the Cap Evaluation that is not contained in the residential sampling version. These QAPP documents identify the data quality objectives (DQOs), analytical requirements, and quality assurance/quality control (QA/QC) procedures that will be implemented to generate defensible data.

This related information is not repeated in the body of this work plan; rather, it is contained in the respective appendices. The HSP Addendum is included in Appendix A, the QAPP Addendum in Appendix B, and the Standard Operating Procedure for the Gravel Sample Collection and Processing is contained Appendix C.

1.1 Objectives of the Evaluation

The primary objectives of the evaluation are to:

- Document current Main Site topography and physical features
- Document the thickness of the gravel (the "Cap"), soil fill materials likely present beneath the Cap (the "Fill"), and clay cover present beneath the Fill (the "Cover") materials present across 22 acres of the Main Site
- Assess the physical content and chemical characteristics of the Cap, Fill, and Cover
- Evaluate the chemical composition of the sampled materials against applicable regulatory criteria
- Prepare a Main Site Evaluation Report to document the findings of the evaluation.

1.2 Project Organization

Following USEPA approval, CH2M HILL will be the lead engineer responsible for implementing the evaluation proposed within this work plan under the direction of Honeywell. Communications will occur regularly among Honeywell, CH2M HILL and USEPA, with the following key points of contact as follows:

- USEPA Remedial Project Manager – Ms. Jena Sleboda
- Honeywell Remediation Manager – Mr. Chuck Geadelmann
- CH2M HILL Project Manager – Mr. Joel Wipf

EPA has primary responsibility for community involvement at the Main Site. Honeywell will provide support to EPA as requested by the Remedial Project Manager (RPM) and Community Involvement Coordinator (CIC) and will coordinate activities through EPA's RPM and CIC.

1.3 Organization of the Work Plan

This Main Site Evaluation Work Plan is organized as follows:

Section 1, Introduction, introduces the Main Site, identifies the work plan components, describes the objectives of the evaluation, and outlines the project and work plan organization.

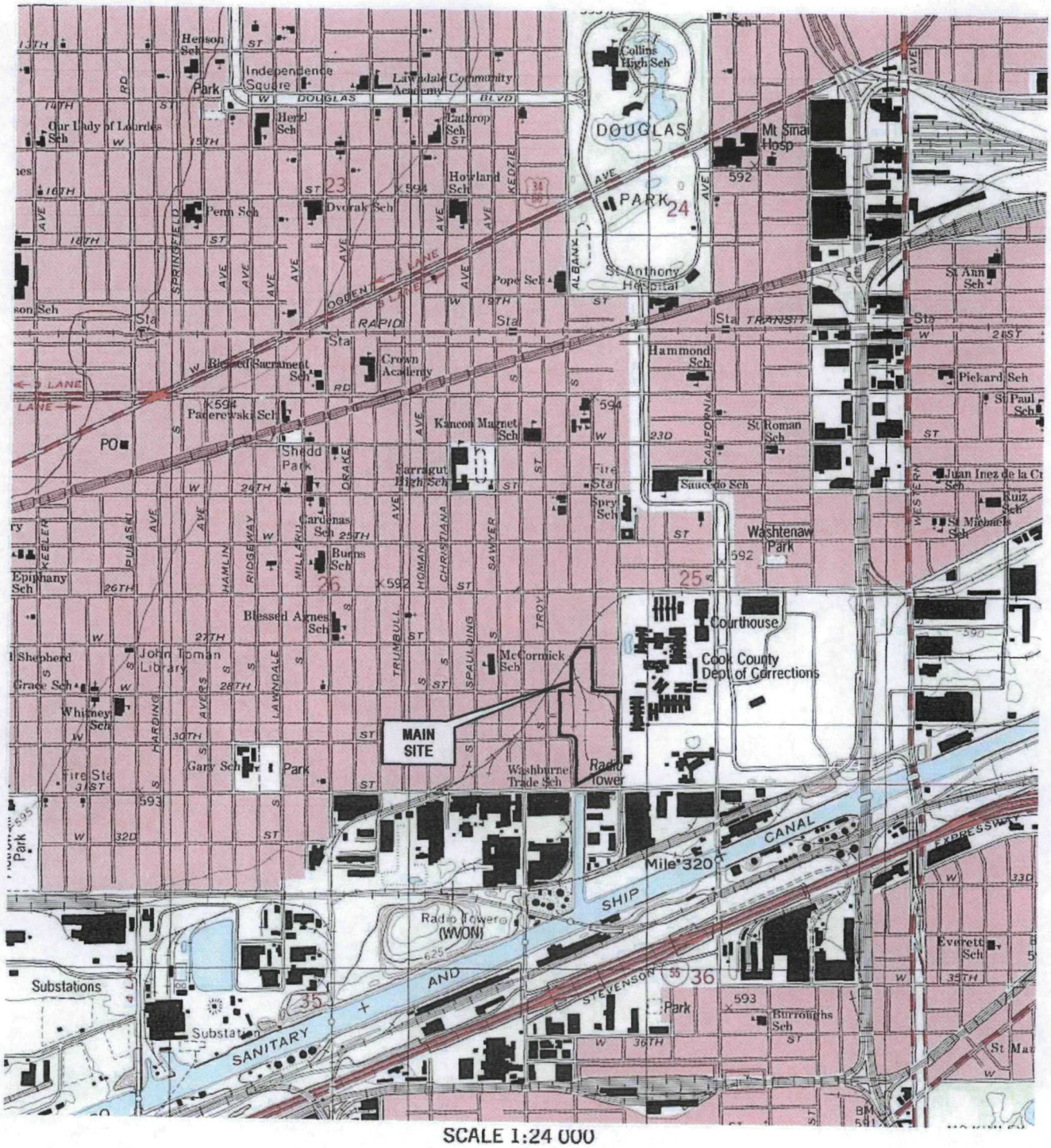
Section 2, Main Site Setting and History, provides an overview of the physical site setting and Main Site history, including certain past operations, previous investigations, and subsequent site activities.

Section 3, Evaluation Rationale and Investigation Procedures, identifies the objectives and describes the proposed cap evaluation procedures.

Section 4, Main Site Evaluation Report, presents the general outline of the evaluation report.

Section 5, Project Schedule, presents the anticipated evaluation schedule based on the scope of the project, and identifies key activities and delivery dates.

Section 6, References, presents a listing of works referenced during compilation of the Main Site Evaluation Work Plan.

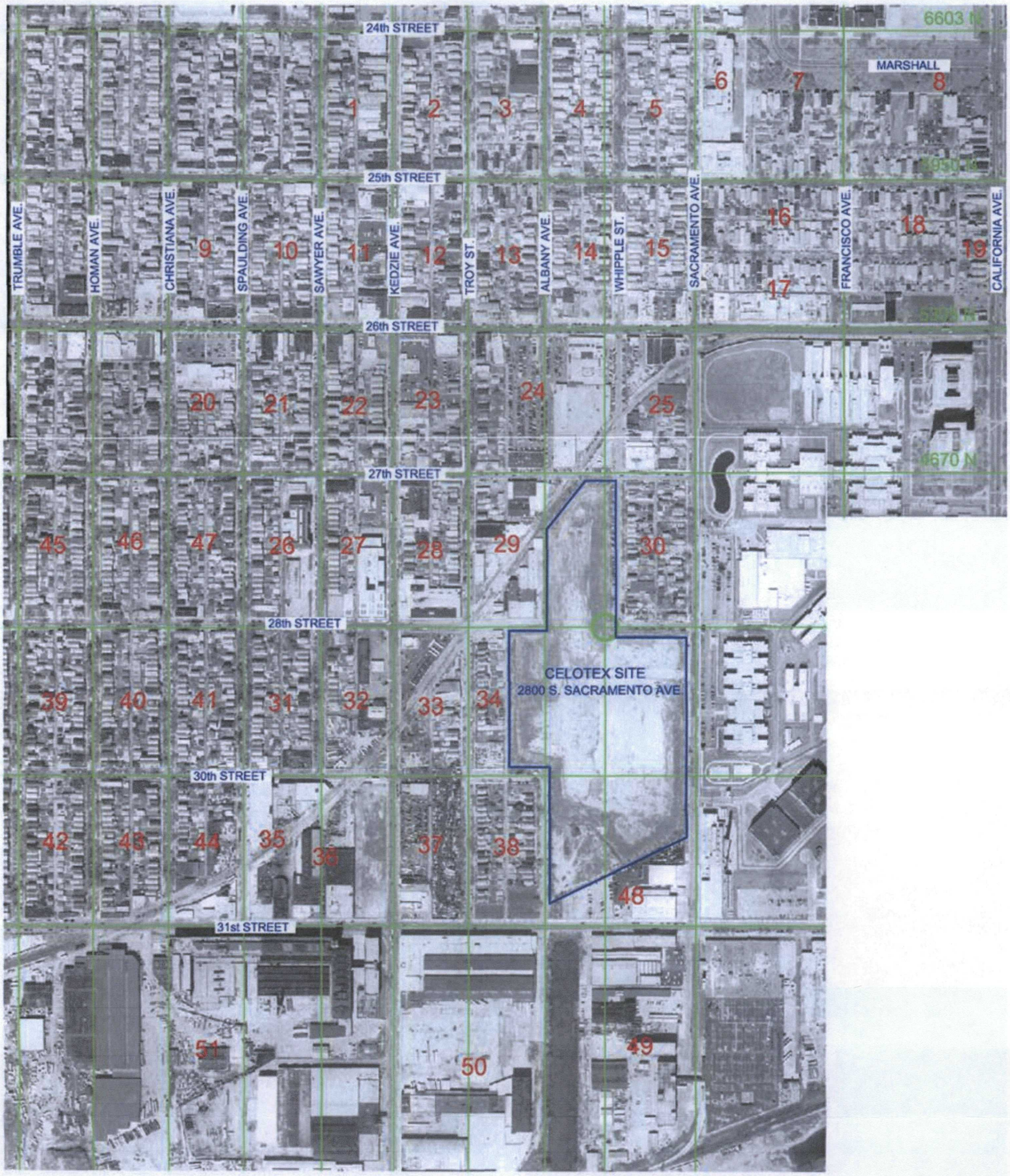


Source: U.S.G.S. 7.5-Minute Quadrangle for Englewood, Illinois, 1997

E327757.CE.10.1 Fig 1-1_Celotex_DRAFT_030906_v5 06-19-06 lg/js

Figure 1-1
Main Site Location
Main Site Evaluation Work Plan
Former Celotex Site
Chicago, Illinois

CH2MHILL



LEGEND

27	Block Number
	Northing and Easting Lines
	Main Site

0 200 400
SCALE: 1"=200'

Figure 1-2
Aerial Photograph
Main Site Evaluation Work Plan
Former Celotex Site
Chicago, Illinois

E327757.CE.10.1 Fig_1-2_Celotex_DRAFT_030906_v5 06-15-06 jls

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SECTION 2

Main Site Setting and History

This section summarizes relevant aspects of the Main Site setting and history based on review of currently available documentation related to past site activities.

2.1 Main Site Setting

The former Celotex Main Site consists of a 22-acre parcel currently owned by 2600 Sacramento Corporation, and a 2-acre parcel currently owned by Monarch Asphalt (Monarch). The United States Geological Survey (USGS) reference for the Main Site location indicates that it is situated in the West 1/2 of the Southwest 1/4 of Section 25, Township 39 North, Range 13 East of the Third Prime Meridian on the Englewood 7.5 Minute Quadrangle.

The Main Site is situated in a multi-use area that includes residential, commercial, manufacturing, governmental, and industrial establishments. The Cook County Correctional Facility is located east of the Main Site on the east side of Sacramento Avenue and the former Atkinson, Topeka & Santa Fe railroad line crosses a portion of the area to the northwest. Residential and commercial properties are located north, west and northeast of the Main Site and industrial property is located to the south. The Chicago Sanitary and Ship Canal is located approximately 1,500 feet south of the Main Site.

The Main Site elevations and topography have not been verified subsequent to the placement of the Fill, and Cap materials on the Main Site. The top of the site is uniformly graded with side slopes around the perimeter. Few other permanent features are present. The site is fenced at the base of the side slopes with a main gate on South Sacramento Avenue near 28th Street.

2.2 Main Site History

The Main Site was used for making, storing and selling asphalt-roofing products. Former operations at the 24-acre Main Site during the approximate period of 1911 to 1989 may have resulted in the release of polycyclic aromatic hydrocarbons (PAHs) to the ground and into the air. Facility closure (1989), demolition of the Main Site (1993), and subsequent actions have all taken place and it has been determined that there are no known ongoing releases, associated with historical operations, occurring from the Main Site.

Currently, the Main Site is elevated compared to surrounding grade due to the presence of Cover, Fill, and Cap materials placed at the site following facility demolition. The placement of materials on the Main Site following demolition post-dates Honeywell's ownership of the Main Site by many years. Honeywell has very little verified information concerning the volume, content, and source of such materials. Based on a review of the limited documentation concerning the materials, it appears that three different types of materials may have been placed over 22 acres of the Main Site (such materials were not placed on the 2-acre Monarch parcel).

First, following completion of facility demolition (during which crushed building materials may have been used as site fill), an approximately 2-foot thick clay cover (the "Cover") was placed over the 22 acre parcel. The source of the clay has not been verified; however, a letter from ERM to USEPA indicates it was undisturbed material generated during a construction project at the Metropolitan Water Reclamation District of Greater Chicago (MWRDGC) wastewater treatment plant in Stickney, Illinois. A site survey documenting the site topography after Cover placement was prepared in 1996 by Westshore Engineering and Surveying for ERM.

Second, miscellaneous fill from other sources (the "Fill"), including possibly fill from a construction project at the Cook County Jail, was likely placed on top of the Cover. Honeywell does not have information concerning when the Fill placement activities took place or the precise thickness of the Fill. It is Honeywell's understanding that the Fill was placed on the Site by the Celotex Corporation. In 1997, following placement of the Fill materials, re-grading of the Main Site was conducted in accordance with a Storm Water Management Plan to address storm water runoff issues. Neither the final topography of the Main Site nor the resulting thickness of the Fill were documented before or after the storm water management plan implementation.

Third, in or about 2002, 2600 Sacramento Corporation placed approximately 2 feet of gravel (the "Cap") over the Fill and Cover materials in order to prepare the Main Site for truck staging operations. The precise placement, final thickness, and source of the Cap material are unknown to Honeywell.

Soils beneath the Cover were sampled in connection with an Engineering Evaluation and Cost Analysis (EE/CA) performed by Honeywell pursuant to a prior Administrative Order on Consent. Following the completion of the EE/CA, USEPA issued an Action Memorandum ("March 2005 Action Memorandum") finding that subsurface contaminants should be addressed by the placement of a 2-foot gravel cap on the Main Site (to the extent one was not already in place) and the recording of certain restrictive covenants. Honeywell and USEPA subsequently entered into a second Administrative Order on Consent ("2006 AOC") whereby Honeywell agreed to perform the activities set forth in the March 2005 Action Memorandum. This evaluation is one of the first tasks under the 2006 AOC.

SECTION 3

Evaluation Rationale and Investigation Procedures

This section details the proposed technical approach and investigation methodologies that will be used to perform the cap evaluation at the Main Site. Details regarding health and safety requirements are addressed in the HSP Addendum (Appendix A). Detailed descriptions of analysis procedures and quality assurance protocols are presented in the QAPP Addendum (Appendix B). Sampling methodology and handling are detailed in the Standard Operating Procedure (SOP, Appendix C).

3.1 Evaluation Rationale

Information collected as part of the cap evaluation will be used for the following purposes:

- Document the current topographic conditions and site features,
- Verify the thickness of the Cap, Fill and Cover material across the 22-acre Main Site parcel,
- Evaluate the chemical composition of the sampled materials against applicable regulatory criteria,
- Determine the physical characteristics of the Cap, Fill, and Cover material.

To confirm the Cap, Fill, and Cover thickness, hollow stem auger (HSA) borings will be drilled at a frequency of two locations per 0.5 acre. Cap, Cover and Fill material penetrated and recovered will be collected continuously, logged and screened with a photoionization detector (PID) for a qualitative evaluation of any organic vapors present.

Due to the low topographic relief of the Main Site and the material previously placed below the Cap, significant changes in Cap thickness are not likely to be encountered at the Main Site between the proposed boring locations. Proposed boring locations are shown on Figure 3-1.

Although borings will not be advanced on the perimeter side slopes, the topographic survey will document the physical characteristics of the side slopes for use in future remedy design.

In addition to physical characterization, the HSA borings will also be used to collect Cap, Fill, and Cover material samples for analysis. HSA borings provide an efficient and reliable method to obtain the samples and associated data for this investigation, while limiting site disruption and impact. The small diameter of the HSA boring will limit potential settling of backfilled evaluation locations that are common when excavating test pits.

3.2 Evaluation Activity

Once the current property owner grants site access for the work, the evaluation activities will be scheduled and conducted. The proposed boring locations will be laid out, a utility locate request submitted, and the site survey will be conducted. Each proposed boring location and elevation, and the current features at the Main Site will be documented. The boring locations

will be laid out based on the proposed two samples per half acre frequency and avoiding the side slope areas. Boring locations will be marked in the field using marking paint, survey stakes, and flagging. The selected evaluation boring locations may be adjusted based on Main Site features observed in the field to avoid operations of the Sacramento Corporation, or as needed based on utility or other subsurface features. Soil borings will not be advanced until the utility locate has been completed.

The Cap, Fill, and Cover materials at each boring location will be penetrated with a HSA to the approximate depth of surrounding grade, to the depth which constitutes the bottom of the Cover material, or until Main Site demolition debris is encountered. Based on currently available information this depth could range from at least 2 feet bgs up to 6 feet bgs. Cap, Cover, and Fill material will be logged and collected continuously with a split spoon, slit barrel or equivalent sampler with sufficient diameter so that the gravel fraction of the Cap material will not prevent sample recovery. Once the entire Cap thickness is penetrated, a conventional sized soil sampling device will be used to collect samples of the Cover and Fill materials for physical characterization, in order to reduce the amount of soil cuttings generated by the sampling activity. Once the limits of the boring are reached based on the identified criteria, augering and soil sampling will cease.

Cap, Fill, and Cover material cuttings generated by the HSA will be stockpiled adjacent to each boring location for reuse backfilling the boring locations.

If refusal is encountered at a proposed boring location, a second boring will be attempted within 10-20 feet of the original assuming access and utility clearance allows. If refusal is also encountered at the second location, the boring location will be excluded from the investigation. Any offsets from refusals due to subsurface obstructions will be documented.

Cap, Cover, and Fill material penetrated and recovered will be screened in 2-foot increments with a photoionization detector (PID) as they are generated for a qualitative evaluation of any organic vapors present. The highest PID reading for each two-foot interval will be recorded. The Cap, Cover, and Fill materials encountered will be generally described by the field technician in accordance with the Unified Soil Classification System (USCS). Changes and boundaries in the Cap, Cover, and Fill materials encountered will be documented.

Three composite samples consisting of aliquots from the four borings per acre will be submitted for analysis by an independent, National Environmental Laboratory Accreditation Program (NELAP)-certified laboratory. The composite samples submitted will be representative of the 1) entire Cap material interval, 2) the Cover, and 3) the Fill materials. The 66 composite samples will be analyzed for the following parameters:

- Semivolatile Organic Compounds (SVOCs) by USEPA Method 8270;
- Arsenic beryllium, cadmium, chromium, lead, mercury, nickel, copper, selenium, silver, thallium, and zinc by USEPA SW6000/7000 series methods (using 6010 where appropriate);
- Synthetic Precipitation Leaching Procedure (SPLP) metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) by USEPA Method 1312/6020/7471;
- Pesticides/Herbicides by USEPA Methods 8081 and 8051A, respectively;
- Polychlorinated Biphenyls (PCBs) by USEPA Method 8082.

A discrete sample from the Cap material, the Cover, and the Fill materials with the highest PID reading in each boring will be submitted for analysis by an independent, NELAP-certified laboratory. The 264 discrete samples will be analyzed for Volatile Organic Compounds (VOCs) by USEPA Method 8260. Further details of the analytical methods, quality assurance/quality control (QA/QC) requirements, data validation, and data management are provided in the QAPP and QAPP addendum.

Composite samples of the Cap material submitted for analysis will be collected and processed in the following manner. Cap material size fractions greater than 2 centimeters in diameter will be manually discarded in the field. The gravel-sized fraction (less than 2 centimeters and greater than 2 millimeters in diameter) and fines (less than 2 millimeters in diameter) of the Cap material composite sample will both be submitted to the laboratory for analysis. The laboratory will sieve the submitted sample to separate the fines from the gravel fraction. The gravel-sized fraction will be weighed and not analyzed. The fines will be analyzed and the results will be calculated and reported based on the "total sample" basis. The total sample includes the mass of the sample for the gravel-sized fraction and the sample mass from the fines. The detailed procedures for Cap material sample collection, field and laboratory processing, and analysis are presented in Standard Operating Procedure (SOP) in Appendix C.

When obtaining a sample of Cap material for volatile analysis, a sample aliquot of the material's fines will be collected in the field and placed into a laboratory prepared vial with distilled water. EnCore® samplers will not be used due to the lack of cohesion of the fines likely preventing collection of the required amount of sample, and USEPA Method 5035 preserved vials will not be used due to the likelihood of effervescence of the limestone in the acidic preservative.

Quality assurance/quality control (QA/QC) samples will be collected at the frequency identified in the QAPP and the QAPP Addendum (Appendix B). The sample collection and analysis methods will match those for the corresponding field sample.

After completing each soil boring, the HSAs will be raised so that the bottom of the auger string is approximately 6 inches below the bottom of the Cap and still in the underlying Fill. The boring annulus below the Cap will be filled with the excess cuttings following sample collection, with the remainder filled with bentonite slurry or hydrated bentonite pellets. Additional bentonite will be added as the augers are raised to approximately 6-inches above the bottom of the Cap to form a seal around the annulus above the Fill. The remaining portion of the annulus to the surface will be filled with the clean Cap material brought up by the auger with any excess clean cap material spread out on the ground around the borehole. Additional gravel will be stockpiled onsite during the work to fill in the boring locations if not enough gravel is recovered to backfill the boring to surrounding grade.

3.3 Sampling Equipment Decontamination

The augers and tools used for borings will be decontaminated prior to mobilization to the Main Site and between boring locations. Following completion of the soil borings, the augers and tools will be decontaminated prior to leaving the site. Decontamination will be conducted using a tap water power wash or steam clean and tap water rinse. A temporary decontamination area will be constructed onsite for use during the fieldwork. Decontamination fluids will be containerized onsite prior to characterization and offsite disposal.

3.4 Data Evaluation

Using the physical characterization data from the borings and the Main Site survey, the condition of the Cap material will be evaluated and any areas where the Cap is less than 2 feet thick will be identified. The current survey data will be compared with the 1996 survey to determine the thickness of material moved or placed onsite after the 1996 survey was conducted. In addition, the depth and disposition of the material in the Cap, Cover, and Fill areas will be contoured.

The sample analysis results will be screened against applicable regulatory criteria.

FURNISHED DESCRIPTION: EXHIBIT "A" (20309340)

THAT PART OF BLOCKS 15-16-24 AND 25 OF SUPERIOR COURT COMMISSIONERS PARTITION OF THE WEST HALF OF THE SOUTHWEST QUARTER SECTION 25, TOWNSHIP 39 NORTH, RANGE 13 EAST OF THE 3RD PRINCIPAL MERIDIAN, DESCRIBED AS FOLLOWS:

BEGINNING AT A POINT ON THE SOUTH LINE OF W. 28TH STREET AS OPENED BY THE SUPERIOR COURT OF COOK COUNTY, SAID POINT BEING 154.79 FEET WEST OF THE WEST LINE OF S. SACRAMENTO AVENUE AS OPENED BY THE SUPERIOR COURT OF COOK COUNTY:

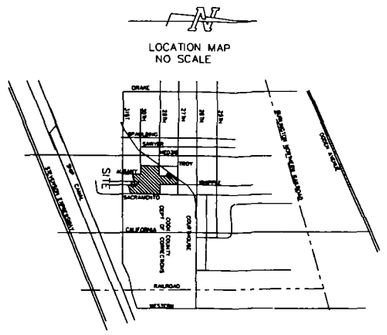
THENCE SOUTH PERPENDICULAR TO SAID SOUTH LINE OF W. 28TH STREET, 54.21 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE, 90.24 FEET,
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 98.85 FEET;
THENCE EAST PERPENDICULAR TO THE LAST DESCRIBED LINE, 60.07 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 127.08 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE, 124.37 FEET;
THENCE, WEST 61.90 FEET ALONG A LINE WHICH FORMS AN ANGLE OF 173 DEGREES 06 MINUTES WITH THE LAST DESCRIBED LINE (MEASURED FROM EAST TO NORTH TO WEST);
THENCE SOUTH 88.64 FEET ALONG A LINE WHICH FORMS AN ANGLE OF 83 DEGREES 02 MINUTES WITH THE LAST DESCRIBED LINE (MEASURED FROM EAST TO SOUTH);
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE, 36.0 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE 101.75 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE 157.89 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 102.69 FEET;
THENCE EAST PERPENDICULAR TO THE LAST DESCRIBED LINE, 33.18 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 68.15 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE, 51.43 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 240.16 FEET;
THENCE EAST PERPENDICULAR TO THE LAST DESCRIBED LINE, 54.30 FEET;
THENCE NORTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 15.50 FEET;
THENCE EAST PERPENDICULAR TO THE LAST DESCRIBED LINE, 97.70 FEET;
THENCE SOUTHEASTERLY 92.65 FEET ALONG A LINE WHICH FORMS AN ANGLE OF 106 DEGREES 10 MINUTES WITH THE LAST DESCRIBED LINE (MEASURED FROM WEST TO SOUTH TO SOUTHEAST);
THENCE EASTERLY 67.09 FEET ALONG A LINE WHICH FORMS AN ANGLE OF 100 DEGREES 07 MINUTES WITH THE LAST DESCRIBED LINE (MEASURED FROM NORTHERLY TO EASTERLY WITH THE LAST DESCRIBED LINE);
THENCE SOUTHEASTERLY 117.30 FEET ALONG A LINE WHICH FORMS AN ANGLE OF 115 DEGREES 02 MINUTES WITH THE LAST DESCRIBED LINE (MEASURED FROM WESTERLY TO SOUTHEASTERLY) TO A POINT ON A LINE, SAID LINE BEING DESCRIBED AS DRAWN FROM A POINT IN THE EAST LINE OF SAID BLOCK 25, SAID POINT BEING 422.06 FEET NORTH OF THE SOUTHEAST CORNER OF SAID BLOCK 25, TO A POINT WHICH IS 83.0 FEET EAST OF THE WEST LINE OF SAID BLOCK 24 AND 133.0 FEET NORTH OF THE SOUTH LINE OF SAID BLOCK 24;
THENCE NORTHEASTERLY ALONG SAID LINE, 311.24 FEET TO THE WEST LINE OF S. SACRAMENTO AVENUE AS OPENED BY THE SUPERIOR COURT OF COOK COUNTY;
THENCE NORTH ALONG SAID WEST LINE OF S. SACRAMENTO AVENUE TO THE SOUTH LINE OF W. 28TH STREET, AS OPENED;
THENCE WEST ALONG SOUTH LINE OF SAID W. 28TH STREET TO THE POINT OF BEGINNING, IN COOK COUNTY, ILLINOIS.

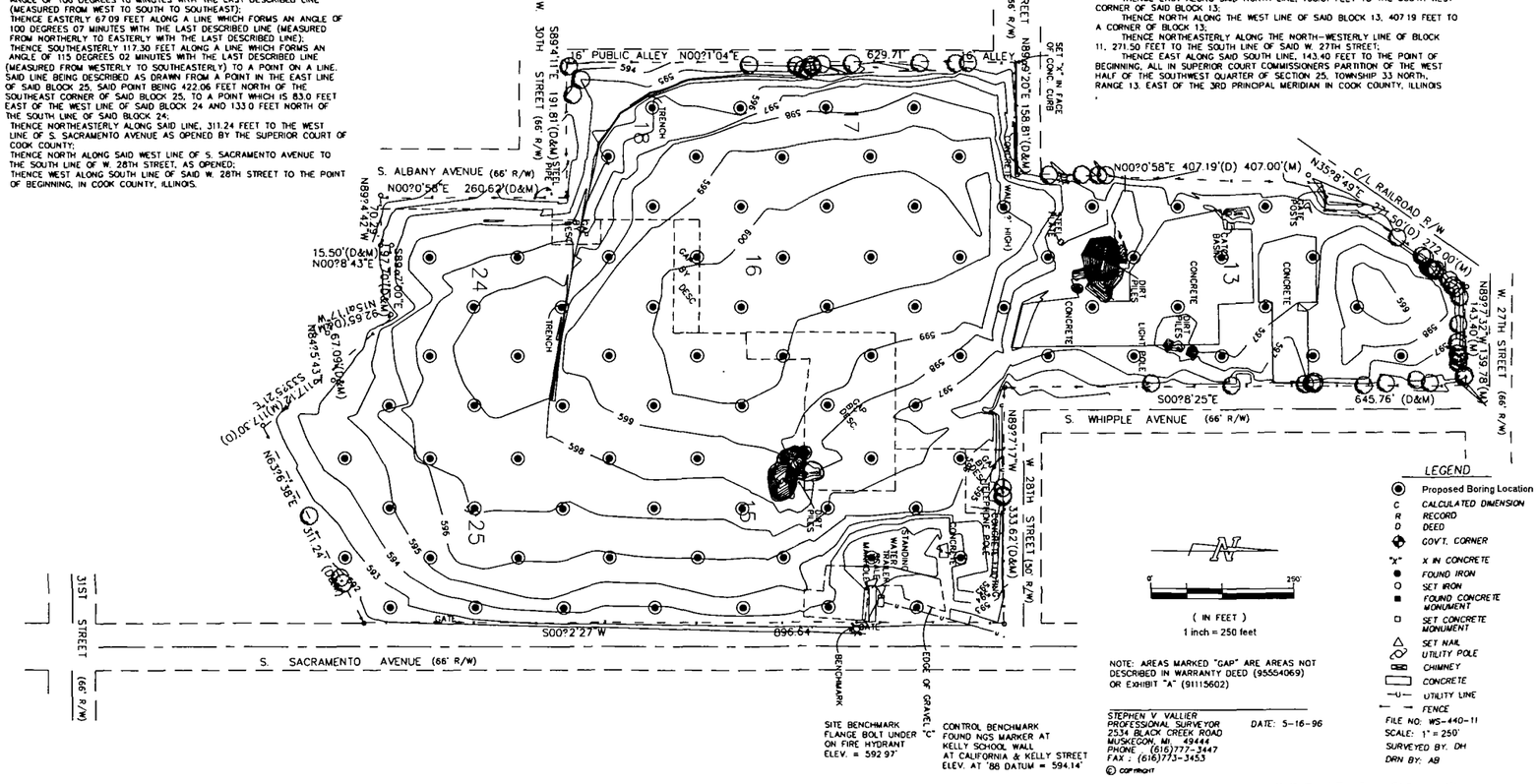
WESTSHORE ENGINEERING & SURVEYING, INC.

TOPOGRAPHIC SURVEY

FOR: ERM NORTH CENTRAL

LOCATION MAP
NO SCALE





0' 250'
(IN FEET)
1 inch = 250 feet

NOTE: AREAS MARKED "GAP" ARE AREAS NOT DESCRIBED IN WARRANTY DEED (95554069) OR EXHIBIT "A" (91115602)

STEPHEN V. VALLIER
PROFESSIONAL SURVEYOR
2534 BLACK CREEK ROAD
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FAX : (616)773-3453

DATE: 5-16-96

© COPYRIGHT

LEGEND

- Proposed Boring Location
- C CALCULATED DIMENSION
- R RECORD
- O DEED
- GOV'T. CORNER
- "X" X IN CONCRETE
- FOUND IRON
- SET IRON
- FOUND CONCRETE MONUMENT
- SET CONCRETE MONUMENT
- SET NAIL
- UTILITY POLE
- CHIMNEY
- CONCRETE
- UTILITY LINE
- FENCE

FILE NO. WS-440-11
SCALE: 1" = 250'
SURVEYED BY: DH
DRN BY: AB

FURNISHED DESCRIPTION: SPECIAL WARRANTY DEED (25237817)

REAL ESTATE IN THE STATE OF ILLINOIS DESCRIBED AS.

BLOCKS 13, 15, 16, 17 AND 18 AND 24, ALL TAKEN AS A TRACT AND DESCRIBED AS FOLLOWS:

BEGINNING AT THE SOUTHWEST CORNER OF W. 27TH STREET AND S. WHIPPLE AVENUE AS OPENED BY THE SUPERIOR COURT OF COOK COUNTY;
THENCE SOUTH ALONG THE WEST LINE OF SAID S. WHIPPLE AVENUE 645.76 FEET TO THE SOUTH LINE OF W. 28TH STREET AS OPENED BY SAID SUPERIOR COURT OF COOK COUNTY;
THENCE EAST ALONG SAID SOUTH LINE, 88.59 FEET TO A POINT 245.03 FEET WEST OF THE WEST LINE OF S. SACRAMENTO AVENUE AS OPENED BY SAID SUPERIOR COURT OF COOK COUNTY;
THENCE SOUTH PERPENDICULAR TO THE SOUTH LINE OF SAID W. 28TH STREET, 151.06 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE 163.69 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 274.64 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE, 157.89 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE 206.16 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE, 18.25 FEET;
THENCE SOUTH PERPENDICULAR TO THE LAST DESCRIBED LINE, 240.16 FEET;
THENCE WEST PERPENDICULAR TO THE LAST DESCRIBED LINE, 16.88 FEET TO A POINT ON THE EAST LINE OF SOUTH ALBANY AVENUE AS DEDICATED BY DOCUMENT NUMBER 4762549;
THENCE NORTH ALONG SAID EAST LINE OF S. ALBANY AVENUE 260.62 FEET TO THE NORTH LINE OF W. 10TH STREET AS PER SAID DEDICATION OF S. ALBANY AVENUE;
THENCE WEST ALONG SAID NORTH LINE OF W. 30TH STREET AS DEDICATED AND SAID NORTH LINE OF W. 30TH STREET AS OPENED BY THE SUPERIOR COURT OF COOK COUNTY, 191.81 FEET TO THE EAST LINE OF PUBLIC ALLEY AS OPENED BY THE SUPERIOR COURT OF COOK COUNTY;
THENCE NORTH ALONG SAID EAST LINE 629.71 FEET TO THE NORTH LINE OF SAID BLOCK 17;
THENCE EAST ALONG SAID NORTH LINE, 158.81 FEET TO THE SOUTH WEST CORNER OF SAID BLOCK 13;
THENCE NORTH ALONG THE WEST LINE OF SAID BLOCK 13, 407.19 FEET TO A CORNER OF BLOCK 13;
THENCE NORTHEASTERLY ALONG THE NORTH-WESTERLY LINE OF BLOCK 11, 271.50 FEET TO THE SOUTH LINE OF SAID W. 27TH STREET;
THENCE EAST ALONG SAID SOUTH LINE, 143.40 FEET TO THE POINT OF BEGINNING, ALL IN SUPERIOR COURT COMMISSIONERS PARTITION OF THE WEST HALF OF THE SOUTHWEST QUARTER OF SECTION 25, TOWNSHIP 39 NORTH, RANGE 13 EAST OF THE 3RD PRINCIPAL MERIDIAN IN COOK COUNTY, ILLINOIS

Figure 3-1

1996 Main Site Survey with Proposed Boring Locations
Main Site Evaluation Work Plan
Former Celotex Site
Chicago, Illinois

SOURCE: Westshore Engineering and Surveying, Inc., 1996, for ERM North Central

SECTION 4

Main Site Evaluation Report

Following data evaluation, a Main Site Evaluation Report will be prepared and submitted to USEPA. A proposed outline of the Main Site Evaluation Report is presented below.

Main Site Evaluation Report Outline

Executive Summary

1. Introduction
   - 1.1 Purpose of Report
   - 1.2 Main Site Background
     - 1.2.1 Main Site Description
     - 1.2.2 Main Site History
     - 1.2.3 Previous Investigations
     - 1.2.4 Physical Setting
   - 1.3 Report Organization

2. Field Activities
   - 2.1 Soil Boring Results
   - 2.2 Main Site Survey
   - 2.3 Sampling and Analysis
   - 2.4 Decontamination of Sampling Equipment

3. Results of Evaluation
   - 3.1 Cap, Cover and Fill Soil Thickness
   - 3.2 Material Observations
   - 3.3 Topographic Update
   - 3.4 Sample Analytical Data
   - 3.5 Data Validation

4. Conclusions and Recommendations

5. References

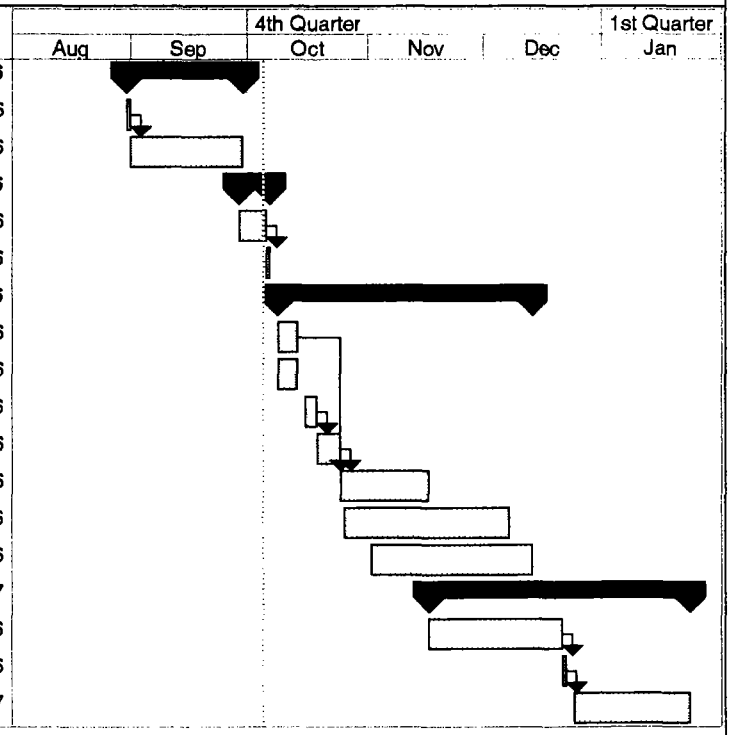
SECTION 5

Project Schedule

Figure 5-1 presents the proposed project schedule for the Main Site Evaluation.

Figure 5-1
Proposed Project Schedule
Main Site Evaluation Work Plan
Former Celotex Site
Chicago, Illinois

ID	Task Name	Duration	Start	Finish
1	**Draft Main Site Evaluation Work Plan**	**30 days**	**Thu 8/31/06**	**Fri 9/29/06**
2	Submit Draft Work Plan to EPA	1 day	Thu 8/31/06	Thu 8/31/06
3	Receive EPA Comments	29 days	Fri 9/1/06	Fri 9/29/06
4	**Final Main Site Evaluation Work Plan**	**8 days**	**Fri 9/29/06**	**Fri 10/6/06**
5	Submit Final Work Plan to EPA	7 days	Fri 9/29/06	Thu 10/5/06
6	Receive Approval From EPA	1 day	Fri 10/6/06	Fri 10/6/06
7	**Implement Main Site Evaluation Work Plan**	**66 days**	**Mon 10/9/06**	**Wed 12/13/06**
8	Notify EPA 5 Days Prior to Start of Field Work	5 days	Mon 10/9/06	Fri 10/13/06
9	Verify or Obtain Access Agreement from Sacramento Corp.	5 days	Mon 10/9/06	Fri 10/13/06
10	Locate Proposed Borings and Perform Site Survey	3 days	Mon 10/16/06	Wed 10/18/06
11	Conduct Utility Clearance	6 days	Thu 10/19/06	Tue 10/24/06
12	Advance Soil Borings, Collect Samples	23 days	Wed 10/25/06	Thu 11/16/06
13	Analyze Soil Samples	43 days	Thu 10/26/06	Thu 12/7/06
14	Data Validation	42 days	Thu 11/2/06	Wed 12/13/06
15	**Prepare Main Site Evaluation Report**	**68 days**	**Fri 11/17/06**	**Tue 1/23/07**
16	Develop Main Site Evaluation Report	35 days	Fri 11/17/06	Thu 12/21/06
17	Submit Main Site Evaluation Report to EPA	1 day	Fri 12/22/06	Fri 12/22/06
18	Receive EPA Comments/Approval	30 days	Mon 12/25/06	Tue 1/23/07



Project: Figure_5-1_Celotex_Schedule_FINAL.mpp
Date: Thu 10/5/06

Task
Split
Progress
Milestone
Summary
Project Summary
External Tasks
External Milestone
Deadline

SECTION 6

References

Environmental Resources Management (ERM)-North Central, Inc., Storm Water Management Plan, Celotex Site, Chicago, Illinois, 1996-1997.

ERM, Letter to USEPA: Celotex Site - Source of Site Cover Material, September 18, 1995.

Parsons Engineering Science, Inc., Data Report for the Engineering Evaluation and Cost Analysis of the 2800 South Sacramento Avenue Site, October 1997.

Parsons Engineering Science, Inc., Engineering Evaluation and Cost Analysis of the 2800 South Sacramento Avenue Site, March 2004.

Westshore Engineering and Surveying, Inc., Topographic Survey for ERM North Central, 1996.

USEPA, Enforcement Action Memorandum, Request for a Non-Time-Critical Removal Action at the 2800 South Sacramento Avenue Site, Chicago, Illinois, March 7, 2005.

APPENDIX A

Health and Safety Plan Addendum

HEALTH AND SAFETY PLAN ADDENDUM
HONEYWELL – FORMER CELOTEX FACILITY
2800 S. SACRAMENTO AVENUE
CHICAGO, ILLINOIS

Job No.: 327757
Prepared by: William M. Berlett, Jr., CIH
Date: March 3, 2006, REV 1 August 29, 2006

HEALTH AND SAFETY PLAN ADDENDUM
Honeywell – Former Celotex Facility
2800 S. Sacramento Avenue
Chicago, Illinois

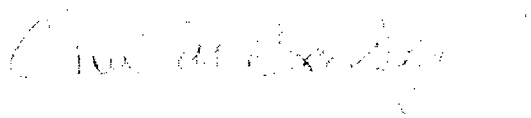
		PHONE
Project Number:	327757	
Project Manager:	Joel Wipf/CHI	773-693-3800x253
Safety Coordinator (SC)	Jim Mallison/CHI	773-693-3800 x202
Honeywell Program H&S Manager (HSM)	Bill Berlett	773-693-3800 x316 847-770-0209 (cell)
Honeywell Remediation Manager	Chuck Geadelmann	763-954-5418
Preparation Date:	March 3, 2006, REV 1 August 29, 2006	
Expiration Date:	August 29, 2007	

APPROVALS

Project Manager:

_____
(DATE)

Honeywell Program Health and Safety Manager:



August 29, 2006
CIH/CSP (DATE)

Safety Coordinator

_____
(DATE)

This Health and Safety Plan is valid only for this specific project as described in Section 3.0. It is not to be used for other projects or subsequent phases of this project without the written approval of the Honeywell Program Health and Safety Manager. **A copy of this plan is to be maintained at the site at all times.**

Health and Safety Plan Addendum

This Health and Safety Plan (HSP) Addendum was prepared to support the evaluation of the cap and cover materials present at the Main Site. This Addendum is an integral part of the HSP prepared for the Residential Soil Sampling Work Plan (CH2M HILL, 2006) and outlines the additions or changes to the HSP associated with the Main Site Activities. This Addendum is to be used in conjunction with the Main Site Cap Evaluation Work Plan (work plan).

INTRODUCTION – SITE BACKGROUND

Revision 1, August 29, 2006 Site background for the Main Site and associated scope of work are as follows:

The Main Site was used for making, storing and selling asphalt-roofing products. Former operations at the 24-acre Main Site during the approximate period of 1911 to 1989 may have resulted in the release of polycyclic aromatic hydrocarbons (PAHs) to the ground and into the air. Facility closure (1989), demolition of the Main Site (1993), and subsequent actions have all taken place and it has been determined that there are no known ongoing releases, associated with historical operations, occurring from the Main Site.

Currently, the Main Site is elevated compared to surrounding grade due to the presence of Cover, Fill, and Cap materials placed at the site following facility demolition. The placement of materials on the Main Site following demolition post-dates Honeywell's ownership of the Main Site by many years. Honeywell has very little verified information concerning the volume, content, and source of such materials. Based on a review of the limited documentation concerning the materials, it appears that three different types of materials may have been placed over 22 acres of the Main Site (such materials where not placed on the 2-acre Monarch parcel).

First, following completion of facility demolition (during which crushed building materials may have been used as site fill), an approximately 2-foot thick clay cover (the "Cover") was placed over the 22 acre parcel. The source of the clay has not been verified; however, a letter from ERM to USEPA indicates it was undisturbed material generated during a construction project at the Metropolitan Water Reclamation District of Greater Chicago (MWRDGC) wastewater treatment plant in Stickney, Illinois. A site survey documenting the site topography after Cover placement was prepared in 1996 by Westshore Engineering and Surveying for ERM.

Second, miscellaneous fill from other sources (the "Fill"), including possibly fill from a construction project at the Cook County Jail, was likely placed on top of the Cover. Honeywell does not have information concerning when the Fill placement activities took place or the precise thickness of the Fill. It is Honeywell's understanding that the Fill was placed on the Site by the Celotex Corporation. In 1997, following placement of the Fill materials, re-grading of the Main Site was conducted in accordance with a Storm Water Management Plan to address storm water runoff issues. Neither the final topography of the Main Site nor the resulting thickness of the Fill were documented before or after the storm water management plan implementation.

Third, in or about 2002, 2600 Sacramento Corporation placed approximately 2 feet of gravel (the "Cap") over the Fill and Cover materials in order to prepare the Main Site for truck staging operations. The precise placement, final thickness, and source of the Cap material are unknown to Honeywell.

The primary objectives of the Main Site evaluation are to:

- Document current Main Site topography and physical features
- Document the thickness of the gravel (the "Cap"), soil fill materials likely present beneath the Cap (the "Fill"), and clay cover present beneath the Fill (the "Cover") materials present across 22 acres of the Main Site
- Assess the physical content and chemical characteristics of the Cap, Fill, and Cover, and
- Evaluate the chemical composition of the sampled materials against applicable regulatory criteria.

Section 1.1.1

The following tasks are regulated under HAZWOPER:

- Residential soil sampling using either a hand auger, power auger, or portable geoprobe drilling machine
- Subsurface investigation on the on-site cap area using hollow stem auger drilling

Section 1.1.2 and 1.1.3

No changes have been made.

Section 1.2

See next page.

1.2 Task Hazard Analysis

TASKS	POTENTIAL HAZARDS (Refer to Hazard Control Section for additional information)																											
	Aerial Lifts	Back Injury (Bending/Lifting)	Biological Hazards	Buried Utilities	Cold Stress	Confined Space Entry	Electrical	Elevated Work Areas/Falls	Entanglement	Excavations	Fires	Flying Debris/Objects	Gas Cylinders	Hand and Power Tools	Heat Stress	Heavy Equipment Exposure	Ionizing Radiation	Lockout-Tagout	Noise	Radio-Frequency Radiation	Respiratory Protection	Slips, Trips and Falls	Stairways and Ladders	Suspended Loads	Traffic Exposure	Vehicle Backing Exposure	Visible Lightning	Working Above or Near Water
Hand/power augering		X	X	X					X			X		X	X				X			X			X			
Portable Geoprobe Machine		X	X	X					X			X		X	X	X			X			X			X			
Property Surveying			X												X							X			X			
Soil sample collection		X	X											X	X							X			X			
Hollow Stem Auger Drilling*		X	X	X					X			X		X	X	X			X			X				X		

Section 1.3

No changes have been made.

Section 2 Hazard Controls and Safe Work Practices

HS&E Plans: CH2M HILL requires HS&E plans for all field projects and subcontractors are required to submit detailed Job Hazard Analysis for their activities as well. The HS&E plan provides a risk analysis of each task and identifies the potential hazards and control measures (including personal protective equipment and air monitoring requirements) for each task.

Job Hazard Analysis (JHAs): JHAs are required by CH2M HILL for all tasks unless the HSM specifically determines it is unnecessary. JHAs provide a step-by-step analysis of the activity being performed and identifies the equipment and control measures necessary to conduct the work safely. JHAs must be reviewed by the work team immediately prior to conducting the work. The JHAs can be a source of information for the daily safety meeting. Copies of JHAs are provided in Attachment 2. Contractors and subcontractors must develop JHAs for their site activities; these must be reviewed by the HSM prior to initiating site activities.

Safety Meetings: CH2M HILL requires that the safety coordinator conduct daily safety meetings to discuss with the field team the task to be performed that day and the potential hazards and mitigation measure. The safety meeting can be used to review the JHA with the team. The Pre Task Safety Plan (PTSP) must be developed each day prior to performing specific work tasks. Each member of the team performing the task must be included in the planning so all are aware of the task hazards and controls. A copy of a PTSP is included in Attachment 11.

Self-Assessments: Project Activity Self-Assessment Checklists are contained in Attachment 3. These checklists provide a method of verifying compliance with established safe work practices, regulations, and industry standards pertaining to hazardous activities. The checklists can be used by any CH2M HILL employee who may be exposed to a hazardous activity or by the SC when providing oversight of a subcontractor performing a hazardous activity. Self-assessments shall be completed prior to subjecting CH2M HILL staff to hazardous operations for any reason. Self-assessment checklists should be completed daily for the first week or until such time that the contractor is exhibiting appropriate work methods, then on a weekly basis thereafter.

If hazardous conditions exist or are apparent during the self-assessment, immediately notify the employees in the area and do not continue work in that area until the conditions are safe. If an imminent danger situation (immediately life threatening or would cause serious injury) exists, immediately stop work, warn all personnel in danger and notify the appropriate safety representative and the CH2M HILL SC. Non-compliance issues identified during the self-assessment shall be immediately rectified. If corrective action assistance is required, the HSM should be contacted for guidance.

Any site-specific requirements outlined in this HS&E Plan that are more stringent than those contained in the self-assessment checklists are to take precedence. The self–assessment checklists are based upon minimum regulatory compliance and some site-specific requirements may be more stringent. The self-assessment checklists, including documented corrective actions, shall be made part of the permanent project records and maintained by the SC.

Site Compliance/Audits:

In order to ensure compliance with requirements contained in the RES H&S Manual, Specification 01620, and with this HASP, audits will be conducted by a HS&E professional as follows: This project shall be audited at least once per year during the duration of the field activities.

Interventions: Honeywell requires that we intervene whenever we see someone exhibiting an unsafe behavior or working in unsafe conditions. When such a situation is observed, an intervention is performed by talking to the person about how the task could be done more safely. Safe Work Observation forms must be completed on a weekly basis, at a minimum, by the SC or FTL. Each completed form must be maintained with the HASP field documents, then transferred to project files upon the completion of the field work. A copy of a Safe Work Observation form is included in Attachment 11.

Sections 2.1 through 3.2

No changes have been made.

Section 3.3

Drug testing for the tasks identified in Section 1 is not required for CH2M HILL employees. If site conditions change and tasks are added contact the HSM to determine if drug testing will be required.

If site conditions and tasks change staff who conduct fieldwork for this project may be required to pass an initial 5-panel drug screen and an alcohol screen within two weeks prior to starting to those applicable field activities. They will be required to enroll in a random testing program for the duration of their work on Honeywell, and will be subject to post-incident and "for cause" testing.

Based on specific work activities/tasks, the drilling contractor-subcontractor personnel will be required to be drug and alcohol screened prior to conducting their field activities. Please contact the Honeywell HSPM for details and to determine if contractor/subcontractor personnel require drug testing.

Section 3.4

No changes have been made.

Section 3.5

Subcontractor: **Drilling contractor TBD**
Subcontractor Safety Representative TBD:
Subcontractor's onsite activities: Hollow stem augering throughout the on-site cap

Section 3.6

No changes have been made.

Section 4.1 PPE Specifications

See next page.

Section 4.1 PPE Specifications

Activity	Level	Body	Head	Respirator [b]
Hand augering	D	Work clothes; steel-toe, leather work boots; leather work gloves; traffic vest if adjacent to roadway.	Hardhat [c] Safety glasses	None anticipated
Power augering/portable direct push drilling	D	Work clothes; steel-toe, leather work boots; leather work gloves; traffic vest if adjacent to roadway .	Hardhat [c] Safety glasses Ear protection [d]	None anticipated
Property surveying	D	Work clothes, leather work shoe	Sunglasses as needed	None required
Soil sample collection Hollow stem drilling	D Modified	Work clothes, coveralls [f] Boots: Leather work boots, may upgrade to include outer rubber boot covers based on site conditions Gloves: Inner surgical-style nitrile & outer chemical-resistant nitrile gloves.	Hardhat [c] Ear protection, as warranted [d]	None anticipated
Tasks requiring upgrade None anticipated, but could be any of the above based on actual site conditions	C	Work clothes or cotton coveralls Boots: Steel-toe leather boots OR steel-toe, leather work boots with outer rubber boot covers Gloves: Inner surgical-style nitrile & outer chemical-resistant nitrile.	Hardhat [c] Splash shield [c] Ear protection [d] Spectacle inserts	APR, full face, with P100 cartridges.

[a] Modifications are as indicated. CH2M HILL will provide PPE only to CH2M HILL employees.
[b] No facial hair that would interfere with respirator fit is permitted.
[c] Hardhat and splash-shield areas are to be determined by the SC.
[d] Ear protection should be worn when conversations cannot be held at distances of 3 feet or less without shouting.
[e] Cartridge change-out schedule will be established by the HSM and at a minimum shall be at least every 8 hours (or one work day), except if relative humidity is > 85%, or if organic vapor measurements are > midpoint of Level C range (refer to Section 5)--then at least every 4 hours. If encountered conditions are different than those anticipated in this HS&E Plan, contact the HSM.

[f] Type of coveralls to be determined by the SC based on actual site conditions.

Section 4.2

No changes have been made.

Section 5.1 Air Monitoring Specifications

Instrument	Tasks	Action Levels[a]		Frequency [b]	Calibration
Photoionization Detector: OVM with 10.6eV lamp or equivalent	All	ND-1 ppm 1-10 ppm If readings exceed 1 ppm, benzene monitoring shall commence	Level D Level C	Initially and periodically during task	Daily
Colorimetric Tube: Drager or equivalent benzene specific 0.5/c (0.5 to 10 ppm range) with pre-tube, or equivalent	All	<0.5 ppm 0.5-1 ppm >1 ppm	Level D Level C Level B	Initially and periodically when PID >1 ppm	Not applicable
Dust Monitor: Miniram model PDM-3 or equivalent	All	0 -3 mg/m^3 > 3 mg/m^3	Level D Level C	Initially and periodically during tasks	Zero Daily
Noise-Level : Auditory	All	Conversations can be held at distances of 3 feet without shouting Conversations cannot be held at a distances of 3 feet without shouting	No action required Hearing protection required	Initially and periodically during task	NA

[a] Action levels apply to sustained (3 minutes or longer) breathing-zone measurements above background.
[b] The exact frequency of monitoring depends on field conditions and is to be determined by the SC; generally, every 5 to 15 minutes if acceptable; more frequently may be appropriate. Monitoring results should be recorded. Documentation should include instrument and calibration information, time, measurement results, personnel monitored, and place/location where measurement is taken (e.g., "Breathing Zone/MW-3," "at surface/SB-2," etc.).
[c] Noise monitoring shall be used at the discretion of the SC.

Sections 5.2 through 12

No changes have been made.

Section 14 Attachments

Attachment 11: Behavior Based Loss Prevention Safety (BBLPS) Forms

Attachment 12: Biological Hazards

APPENDIX B

Quality Assurance Project Plan Addendum

QUALITY ASSURANCE PROJECT PLAN

ADDENDUM

For the
Former Celotex Site
2800 South Sacramento Avenue
Chicago, Illinois 60623

Prepared for

Honeywell International Inc.

October 2006

Prepared by

CH2MHILL

QUALITY ASSURANCE PROJECT PLAN – Addendum

RESIDENTIAL STUDY AREA NEAR THE FORMER CELOTEX SITE
Chicago, Illinois

Honeywell International Inc.

Prepared by: CH2M HILL

Date: ~~June~~ Oct. 2006

Approved by:

USEPA, Region 5, Remedial Project Manager
~~Rosita Clarke-Moreno~~ JENA SLEBODA

USEPA, Region 5, Quality Assurance Reviewer

CH2M HILL Project Manager
Joel Wipf

CH2M HILL Quality Assurance Manager

Laboratory Operations Manager

Quality Assurance Officer

Quality Assurance Project Plan Addendum

This Quality Assurance Project Plan (QAPP) Addendum was prepared to support the Main Site Evaluation for the former Celotex site located in Chicago, Illinois. This Addendum is an integral part of the QAPP prepared for the Residential Soil Sampling Work Plan (CH2M HILL, June 2006) and outlines the additions or changes to the QAPP, which are specific to the Main Site activities. This Addendum is to be used in conjunction with the Main Site Evaluation Work Plan (work plan) and the Sample Collection and Handling of Cap Material at the Honeywell Celotex Site Standard Operating Procedure (SOP) included as Appendix C to the work plan (CH2M HILL, August 2006).

Sections 1.1 through 1.5.1

No changes have been made.

Section 1.5.2—Project Schedule

The sampling schedule will be determined by the project manager and/or field team leader. The laboratory will need to make arrangements to accept deliveries as needed, take custody of the samples, and set aside production capacity. This task will involve the collection of both composite samples and discrete samples. Additional QA/QC samples will also be collected. The target parameter lists and reporting limits can be found in Table 2 through Table 8.

Sections 1.6 through 1.8

No changes have been made.

Section 2.1—Sample Design

Section 2.1.1—Soil Sampling Summary

Refer to the sample collection SOP for detailed descriptions of the field procedures that will be used. Surface and subsurface soil samples will be collected. An estimated 66 composite samples and additional field QC samples will be collected. These samples will be analyzed for Semi-volatile Organic Compounds (SVOCs) SW-846 method 8270, Pesticides SW-846 method 8081, Polychlorinated Biphenyls (PCBs) SW-846 method 8082, Herbicides SW-846 method 8151, Metals (As, Be, Cd, Cr, Pb, Hg, Ni, Cu, Se, Ag, Tl, Zn) SW-846 method 6010, and SPLP Metals (As, Ba, Cd, Cr, Pb, Hg, Se, Ag) SW-846 method 1312/6010. In addition, an estimated 264 discrete samples, along with associated QC samples, will be collected and analyzed for Volatile Organic Compounds (VOCs) SW-846 method 8260.

Section 2.1.2—Sampling Method Requirements

Refer to the work plan and sample collection SOP for sampling procedures. Also, the work plan provides detailed information regarding decontamination of field equipment.

Section 2.2—Sample Handling and Custody Requirements

Section 2.2.1—Sample Handling and Preservation

Refer to section 2.2.1 of the QAPP for additional information. Further details can also be found in the work plan and sample collection SOP. Sample containers and preservation requirements specific to this field event are listed in Table 1 of the QAPP addendum. The lab performing the analysis may specify different sample containers and volumes than those listed in the table.

Sections 2.2.2 through 2.2.4.7

No changes have been made.

Section 2.3—Analytical Method Requirements

Section 2.3.1—Target Analytes and Reporting Limits

Refer to section 2.3.1 of the QAPP for additional information. Target parameter lists and reporting limits can be found in Table 2 through Table 8 of the QAPP addendum.

Sections 2.3.2 through 2.4.1.1

No changes have been made.

Section 2.4.1.2—Quality Control Analysis Originated by the Field Team

Refer to section 2.4.1.2 of the QAPP for additional information. Laboratory QC requirements for each analytical method can be found in Table 2 through Table 8 of the QAPP addendum.

Section 2.4.2

No changes have been made.

Sections 2.5 through 4.3

No changes have been made.

Tables

- **Table 1**—required Analytical Methods, Sample Containers, Preservation, and Holding Times

- **Table 2 through Table 8**—Target Parameter Lists / Reporting Limits and QC Requirements

Appendixes

Analytical SOPs for the parameters identified in Section 2.1.1 are contained in Appendix A.

TABLE 1
Required Analytical Method, Sample Containers, Preservation, and Holding Times

Analyses	Analytical Method	Sample Matrix [a]	Container [b]	Qty	Preservative [c]	Holding Time [d]
Volatile Organic Compounds	SW-846 5030B/8260B	W	40 mL, glass	3	HCL, pH<2, cool to 4 °C	14 days
	SW-846 5035/8260B	S	5g – EZ-draw syringe, 40 mL, glass	3	Water, Cool 4°C	48 hours from collection to preservation, 14 days to analysis
			5g - EZ-draw syringe, 40 mL, glass	1	Methanol, Cool 4°C	
			2 oz, glass	1	Cool 4°C	
Semi-volatile Organic Compounds	SW-846 3510C/3520C/8270C	W	1-L amber glass	2	Cool 4°C	7/40 days[e]
	SW-846 3550B/8270C	S	8-oz glass	1	Cool 4°C	14/40 days[f]
Organochlorine Pesticides	SW-846 3510C/3520C/8081A	W	1-L amber glass	2	Cool 4°C	7/40 days[e]
	SW-846 3550B/8081A Cleanup – 3620B	S	8-oz glass	1	Cool 4°C	14/40 days[f]
Polychlorinated Biphenyls	SW-846 3510C/3520C/8082	W	1-L amber glass	2	Cool 4°C	7/40 days[e]
	SW-846 3550B/8082 Cleanup – 3665A	S	8-oz glass	1	Cool 4°C	14/40 days[f]
Herbicides	SW-846 3510C/8151A	W	1-L amber glass	2	Cool 4°C	7/40 days[e]
	SW-846 3550B/8151A	S	8-oz glass	1	Cool 4°C	14/40 days[f]
Metals (Total)	SW-846 3010A/3020A-SW6010B /7000 Series	W	500-mL polyethylene	1	HNO_3, pH < 2Cool 4°C	6 months; Mercury 28 days
	SW-846 3050-SW6010B/7000 Series	S	8-oz glass	1	Cool 4°C	
SPLP–Metals (Total)	SW-846 1312/6020/7000 Series	W	500-mL polyethylene	1	HNO_3, pH < 2Cool 4°C	6/6 months[g]
		S	8-oz glass	1	Cool 4°C	28/28 days[g]

Notes: Sample container, and volume requirements will be specified by the analytical laboratory performing the tests.
Three times the required volume should be collected for samples designated as MS/MSD samples.

[a] Sample matrix: S = surface soil, subsurface soil, sediment; W = surface water
[b] All containers will be sealed with Teflon®-lined screw caps.
[c] All samples will be stored promptly at 4°C in an insulated chest.
[d] Holding times are from the time of sample collection
[e] 7 days to extraction for water, 40 days for analysis
[f] 14 days to extraction for soil, 40 days for analysis.
[g] 6 months to extraction, 6 months to analysis, except Mercury – 28 days to extraction, 28 days to analysis...

Source: SW-846, third edition, Update III (June 1997).
°C = degrees Centigrade; g = gram; HCl = hydrochloric acid; L = liter; TCLP = synthetic precipitation leaching procedure; oz = ounce; mL = milliliter; HNO_3 = nitric acid

TABLE 2.1
RLs for Method SW8260B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
SW-846 8260B	1,1,1-Trichloroethane	1	µg/L	5	µg/Kg
VOCs	1,1,2,2-Tetrachloroethane	1	µg/L	5	µg/Kg
	1,1,2-Trichloroethane	1	µg/L	5	µg/Kg
	1,1,2-Trichloro-1,2,2-trifluoroethane	1	µg/L	5	µg/Kg
	1,1-Dichloroethane	1	µg/L	5	µg/Kg
	1,1-Dichloroethene	1	µg/L	5	µg/Kg
	1,2,4-Trichlorobenzene	1	µg/L	5	µg/Kg
	1,2-Dichloroethane	1	µg/L	5	µg/Kg
	1,2-Dichlorobenzene	1	µg/L	5	µg/Kg
	1,2-Dibromo-3-chloropropane	2	µg/L	5	µg/Kg
	1,2-Dichloropropane	1	µg/L	5	µg/Kg
	1,2-Dibromoethane (EDB)	1	µg/L	5	µg/Kg
	1,3-Dichlorobenzene	1	µg/L	5	µg/Kg
	1,4-Dichlorobenzene	1	µg/L	5	µg/Kg
	2-Butanone (MEK)	5	µg/L	10	µg/Kg
	2-Hexanone	5	µg/L	5	µg/Kg
	4-methyl-2-pentanone	5	µg/L	10	µg/Kg
	Acetone	5	µg/L	10	µg/Kg
	Benzene	1	µg/L	5	µg/Kg
	Bromodichloromethane	1	µg/L	5	µg/Kg
	Bromoform	1	µg/L	5	µg/Kg
	Bromomethane	1	µg/L	5	µg/Kg
	Carbon disulfide	1	µg/L	5	µg/Kg
	Carbon tetrachloride	1	µg/L	5	µg/Kg
	Chlorobenzene	1	µg/L	5	µg/Kg
	Chloroethane	1	µg/L	5	µg/Kg
	Chloroform	1	µg/L	5	µg/Kg
	Chloromethane	1	µg/L	5	µg/Kg
	cis-1,2-DCE	1	µg/L	5	µg/Kg
	cis-1,3-Dichloropropene	1	µg/L	5	µg/Kg

TABLE 2.1
RLs for Method SW8260B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
	Cyclohexane	1	µg/L	5	µg/Kg
	Dibromochloromethane	1	µg/L	5	µg/Kg
	Dichlorodifluoromethane	1	µg/L	5	µg/Kg
	Ethylbenzene	1	µg/L	5	µg/Kg
	Isopropylbenzene	1	µg/L	5	µg/Kg
	Methyl acetate	2	µg/L	5	µg/Kg
	Methylcyclohexane	1	µg/L	5	µg/Kg
	Methylene chloride	2	µg/L	5	µg/Kg
	Styrene	1	µg/L	5	µg/Kg
	Trichloroethene	1	µg/L	5	µg/Kg
	tert-Butyl Methyl Ether	1	µg/L	5	µg/Kg
	Tetrachloroethene	1	µg/L	5	µg/Kg
	Toluene	1	µg/L	5	µg/Kg
	trans-1,2-Dichloroethene	1	µg/L	5	µg/Kg
	trans-1,3-Dichloropropene	1	µg/L	5	µg/Kg
	Trichlorofluoromethane	1	µg/L	5	µg/Kg
	Vinyl Chloride	1	µg/L	5	µg/Kg
	Xylenes (total)	3	µg/L	5	µg/Kg

TABLE 2.2
QC Acceptance Criteria for Method SW8260B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS
SW-846 8260B	1,1,1-Trichloroethane	67-132	≤ 20	68-130	≤ 30	1
VOCs	1,1,2,2-Tetrachloroethane	63-128	≤ 20	59-140	≤ 30	3
	1,1,2-Trichloroethane	75-125	≤ 20	62-127	≤ 30	1
	1,1,2-Trichloro-1,2,2-trifluoroethane	70-130	≤ 20	70-130	≤ 30	1
	1,1-Dichloroethane	69-133	≤ 20	73-125	≤ 30	1
	1,1-Dichloroethene	68-130	≤ 20	65-136	≤ 30	1
	1,2,4-Trichlorobenzene	66-134	≤ 20	65-131	≤ 30	3
	1,2-Dichloroethane	69-132	≤ 20	72-137	≤ 30	1
	1,2-Dichlorobenzene	71-122	≤ 20	74-120	≤ 30	3
	1,2-Dibromo-3-chloropropane	50-132	≤ 20	49–135	≤ 30	3
	1,2-Dichloropropane	65-125	≤ 20	65-120	≤ 30	1
	1,2-Dibromoethane	80-121	≤ 20	70-124	≤ 30	2
	1,3-Dichlorobenzene	75-124	≤ 20	72-124	≤ 30	3
	1,4-Dichlorobenzene	74-123	≤ 20	72-125	≤ 30	3
	2-Butanone	49-136	≤ 20	40-135	≤ 30	1
	2-Hexanone	65-135	≤ 20	60-140	≤ 30	2
	4-methyl-2-pentanone	65-135	≤ 20	60-140	≤ 30	1
	Acetone	40-135	≤ 20	40-141	≤ 30	1
	Benzene	75-125	≤ 20	73-126	≤ 30	1
	Bromodichloromethane	76-121	≤ 20	72-128	≤ 30	1
	Bromoform	69-128	≤ 20	66-137	≤ 30	2
	Bromomethane	53-141	≤ 20	45-141	≤ 30	1
	Carbon disulfide	70-130	≤ 20	65-135	≤ 30	2
	Carbon tetrachloride	66-138	≤ 20	67-133	≤ 30	1
	Chlorobenzene	75-125	≤ 20	75-123	≤ 30	2
	Chloroethane	58-133	≤ 20	41-141	≤ 30	1
	Chloroform	69-128	≤ 20	72-124	≤ 30	1
	Chloromethane	56-131	≤ 20	51-129	≤ 30	1
	Cis-1,2-Dichloroethene	72-126	≤ 20	67-125	≤ 30	1

TABLE 2.2
QC Acceptance Criteria for Method SW8260B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS
	Cis-1,3-Dichloropropene	69-131	≤ 20	72-126	≤ 30	1
	Cyclohexane	70-130	≤ 20	70-130	≤ 30	1
	Dibromochloromethane	66-133	≤ 20	66-130	≤ 30	2
	Dichlorodifluoromethane	53-153	≤ 20	34-136	≤ 30	1
	Ethylbenzene	70-130	≤ 20	70-130	≤ 30	1
	Isopropylbenzene	75-127	≤ 20	77-129	≤ 30	3
	Methyl acetate	70-130	≤ 20	70-130	≤ 30	1
	Methylcyclohexane	70-130	≤ 20	70-130	≤ 30	2
	Methylene chloride	63-137	≤ 20	63-137	≤ 30	1
	Styrene	65-134	≤ 20	74-128	≤ 30	2
	Trichloroethene	70-127	≤ 20	77-124	≤ 30	1
	Trichlorofluoromethane	57-129	≤ 20	49-139	≤ 30	1
	tert-Butyl Methyl Ether	65-123	≤ 20	50-135	≤ 30	1
	Tetrachloroethene	66-128	≤ 20	67-139	≤ 30	2
	Toluene	77-122	≤ 20	71-127	≤ 30	1
	trans-1,2-Dichloroethene	63-137	≤ 20	66-134	≤ 30	1
	trans-1,3-Dichloropropene	59-135	≤ 20	65-127	≤ 30	1
	Vinyl Chloride	50-134	≤ 20	58-126	≤ 30	1
	Xylenes (total)	70-130	≤ 20	70-130	≤ 30	3
	Surrogates:					
	Dibromofluoromethane	85-115		65–135		
	Toluene-D8	81-120		84-116		
	4-Bromofluorobenzene	76-119		84-118		
	1,2-DCA-D4	72-119		52–149		
	Internal Standards:					
	Fluorobenzene					1
	Chlorobenzene-D5					2
	1,4-Dichlorobenzend-D					3

TABLE 3.1
RLs for Method SW8270C

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
SW-846 8270C	1,1'-Biphenyl	10	µg/L	330	µg/Kg
Semivolatile organics	2,2'-oxybis(1-Chloropropane)	10	µg/L	330	µg/Kg
Base/Neutral Extractables	2,4-Dinitrotoluene	10	µg/L	330	µg/Kg
	2,6-Dinitrotoluene	10	µg/L	330	µg/Kg
	2-Chloronaphthalene	10	µg/L	330	µg/Kg
	2-Methylnaphthalene	10	µg/L	330	µg/Kg
	2-Nitroaniline	25	µg/L	830	µg/Kg
	3-Nitroaniline	25	µg/L	830	µg/Kg
	3,3-Dichlorobenzidine	10	µg/L	330	µg/Kg
	4-Bromophenyl phenyl ether	10	µg/L	330	µg/Kg
	4-Chlorophenyl phenyl ether	10	µg/L	330	µg/Kg
	4-Chloroaniline	10	µg/L	330	µg/Kg
	4-Nitroaniline	25	µg/L	830	µg/Kg
	Acenaphthylene	10	µg/L	330	µg/Kg
	Acenapthene	10	µg/L	330	µg/Kg
	Acetophenone	10	µg/L	330	µg/Kg
	Atrazine	10	µg/L	330	µg/Kg
	Anthracene	10	µg/L	330	µg/Kg
	Benzo (a) anthracene	10	µg/L	330	µg/Kg
	Benzo (a) pyrene	10	µg/L	330	µg/Kg
	Benzo (k) fluoranthene	10	µg/L	330	µg/Kg
	Benzo (b) fluoranthene	10	µg/L	330	µg/Kg
	Benzo (g,h,i) perylene	10	µg/L	330	µg/Kg
	Bis (2-chloroethoxy) methane	10	µg/L	330	µg/Kg
	Bis (2-chloroethyl) ether	10	µg/L	330	µg/Kg
	Bis (2-ethylhexyl) phthalate	10	µg/L	330	µg/Kg
	Butyl benzylphthalate	10	µg/L	330	µg/Kg
	Benzaldehyde	10	µg/L	330	µg/Kg
	Caprolactam	10	µg/L	330	µg/Kg

TABLE 3.1
RLs for Method SW8270C

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
	Carbazole	10	µg/L	330	µg/Kg
	Chrysene	10	µg/L	330	µg/Kg
	Di-n-butylphthalate	10	µg/L	330	µg/Kg
	Di-n-octylphthalate	10	µg/L	330	µg/Kg
	Dibenz (a,h) anthracene	10	µg/L	330	µg/Kg
	Dibenzofuran	10	µg/L	330	µg/Kg
	Diethyl phthalate	10	µg/L	330	µg/Kg
	Dimethyl phthalate	10	µg/L	330	µg/Kg
	Fluoranthene	10	µg/L	330	µg/Kg
	Fluorene	10	µg/L	330	µg/Kg
	Hexachlorobenzene	10	µg/L	330	µg/Kg
	Hexachlorobutadiene	10	µg/L	330	µg/Kg
	Hexachlorocyclopentadiene	10	µg/L	330	µg/Kg
	Hexachloroethane	10	µg/L	330	µg/Kg
	Indeno (1,2,3-cd) pyrene	10	µg/L	330	µg/Kg
	Isophorone	10	µg/L	330	µg/Kg
	n-Nitrosodiphenylamine	10	µg/L	330	µg/Kg
	n-Nitrosodi-n-propylamine	10	µg/L	330	µg/Kg
	Naphthalene	10	µg/L	330	µg/Kg
	Nitrobenzene	10	µg/L	330	µg/Kg
	Phenanthrene	10	µg/L	330	µg/Kg
	Pyrene	10	µg/L	330	µg/Kg
SW-846 8270C	2,4,5-Trichlorophenol	25	µg/L	830	µg/Kg
Semivolatile organics	2,4,6-Trichlorophenol	10	µg/L	330	µg/Kg
Acid Extractables	2,4-Dichlorophenol	10	µg/L	330	µg/Kg
	2,4-Dimethylphenol	10	µg/L	330	µg/Kg
	2,4-Dinitrophenol	25	µg/L	830	µg/Kg
	2-Chlorophenol	10	µg/L	330	µg/Kg
	2-Methylphenol	10	µg/L	330	µg/Kg
	2-Nitrophenol	10	µg/L	330	µg/Kg

TABLE 3.1
RLs for Method SW8270C

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
	4,6-Dinitro-2-methylphenol	25	µg/L	830	µg/Kg
	4-Chloro-3-methylphenol	10	µg/L	330	µg/Kg
	4-Methylphenol	10	µg/L	330	µg/Kg
	4-Nitrophenol	25	µg/L	830	µg/Kg
	Pentachlorophenol	25	µg/L	830	µg/Kg
	Phenol	10	µg/L	330	µg/Kg

TABLE 3.2
QC Acceptance Criteria for Method SW8270C

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc Sur.
SW-846 8270C	1,1'-Biphenyl	52–120	≤ 20	46–125	≤ 30	3	2
SVOCs	1,2,4-Trichlorobenzene	37–120	≤ 20	44–125	≤ 30	2	4
	2,2'-Oxybis (1-Chloropropane)	50-150	≤ 20	50-150	≤ 30	1	2
	2,4-Dinitrotoluene	51–120	≤ 20	48–125	≤ 30	3	4
	2,6-Dinitrotoluene	49–120	≤ 20	48–125	≤ 30	3	4
	2-Chloronaphthalene	49–120	≤ 20	45–125	≤ 30	3	4
	2-Methylnaphthalene	46–120	≤ 20	47–125	≤ 30	2	5
	2-Nitroaniline	48–120	≤ 20	44–125	≤ 30	3	2
	3-Nitroaniline	20–126	≤ 20	27–125	≤ 30	3	2
	3,3'-Dichlorobenzidine	50-150	≤ 20	50-150	≤ 30	5	5
	4-Bromophenyl phenyl ether	52–120	≤ 20	46–125	≤ 30	4	1
	4-Chlorophenyl phenyl ether	50–120	≤ 20	47–125	≤ 30	3	4
	4-Chloroaniline	50-150	≤ 20	50-150	≤ 30	2	2
	4-Methylphenol	50-150	≤ 20	50-150	≤ 30	1	5
	4-Nitroaniline	36–120	≤ 20	34–125	≤ 30	3	4
	Acenaphthylene	50–120	≤ 20	44–125	≤ 30	3	4
	Acenaphthene	47–120	≤ 20	46–125	≤ 30	3	4
	Acetophenone	50-150	≤ 20	50-150	≤ 30	1	2
	Anthracene	54–120	≤ 20	53–125	≤ 30	4	1
	Atrazine	50-150	≤ 20	50-150	≤ 30	4	4
	Benz (a) anthracene	56–100	≤ 20	52–125	≤ 30	5	6
	Benzo (a) pyrene	53–120	≤ 20	50–125	≤ 30	6	6
	Benzo (b) fluoranthene	45–124	≤ 20	45–125	≤ 30	6	6
	Benzo (g,h,I) perylene	38–123	≤ 20	38–126	≤ 30	6	6
	Benzo (k) fluoranthene	45-124	≤ 20	45-125	≤ 30	6	6
	Bis (2-chloroethoxy) methane	46–120	≤ 20	43–125	≤ 30	2	5
	Bis (2-chloroethyl) ether	37–120	≤ 20	38–125	≤ 30	1	3
	Bis (2-ethylhexyl) phthalate	42–126	≤ 20	47–127	≤ 30	5	6
	Butyl benzyl phthalate	46–120	≤ 20	49–125	≤ 30	5	6

TABLE 3.2
QC Acceptance Criteria for Method SW8270C

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc Sur.
	Benzaldehyde	50-150	≤ 20	50-150	≤ 30	1	2
	Caprolactam	50-150	≤ 20	50-150	≤ 30	2	2
	Carbazole	50-120	≤ 20	40-135	≤ 30	4	4
	Chrysene	55–120	≤ 20	53–125	≤ 30	5	6
	Di-n-butyl phthalate	54–120	≤ 20	56–125	≤ 30	4	1
	Di-n-octyl phthalate	37–137	≤ 20	41–132	≤ 30	5	6
	Dibenz (a,h) anthracene	42–127	≤ 20	41–125	≤ 30	6	6
	Dibenzofuran	54–120	≤ 20	51–125	≤ 30	3	4
	Diethyl phthalate	41–120	≤ 20	50–125	≤ 30	3	4
	Dimethyl phthalate	25–127	≤ 20	49–125	≤ 30	3	4
	Fluoranthene	54–120	≤ 20	54–125	≤ 30	4	1
	Fluorene	50–120	≤ 20	49–125	≤ 30	3	2
	Hexachlorobenzene	52–120	≤ 20	47–125	≤ 30	4	1
	Hexachlorobutadiene	27–120	≤ 20	40–125	≤ 30	2	5
	Hexachloroethane	28–120	≤ 20	34–125	≤ 30	1	3
	Indeno (1,2,3-c,d) pyrene	43–125	≤ 20	38–125	≤ 30	5	6
	Isophorone	50–120	≤ 20	43–125	≤ 30	2	5
SW-846 8270C	n-Nitrosodi-n-propylamine	34–128	≤ 20	40–125	≤ 30	1	3
SVOCs	n-Nitrosodiphenylamine	48–120	≤ 20	49–125	≤ 30	4	1
	Naphthalene	39–120	≤ 20	40–125	≤ 30	2	5
	Nitrobenzene	44–120	≤ 20	41–125	≤ 30	2	4
	Phenanthrene	51–120	≤ 20	50–125	≤ 30	4	1
	Pyrene	49–128	≤ 20	46–125	≤ 30	5	6
	2,4,5-Trichlorophenol	49–120	≤ 20	49–125	≤ 30	3	1
	2,4,6-Trichlorophenol	49–126	≤ 20	43–125	≤ 30	3	1
	2,4-Dichlorophenol	48–120	≤ 20	45–125	≤ 30	2	5
	2,4-Dimethylphenol	28–120	≤ 20	32–125	≤ 30	2	5
	2,4-Dinitrophenol	25–130	≤ 20	25–132	≤ 30	3	4
	2-Chlorophenol	37–120	≤ 20	44–125	≤ 30	1	3

TABLE 3.2
QC Acceptance Criteria for Method SW8270C

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc Sur.
	2-Methylphenol	38–120	≤ 20	40–125	≤ 30	1	3
	2-Nitrophenol	39–123	≤ 20	42–125	≤ 30	2	4
	4,6-Dinitro-2-Methyl Phenol	40–130	≤ 20	29–137	≤ 30	4	1
	4-Chloro-3-Methyl Phenol	47–120	≤ 20	46–125	≤ 30	2	5
	4-Nitrophenol	20–120	≤ 20	25–138	≤ 30	3	2
	Pentachlorophenol	38–120	≤ 20	25–125	≤ 30	4	1
	Phenol	20–120	≤ 20	39–125	≤ 30	1	5
	Surrogates:						
	2,4,6-Tribromophenol	42–124		36–126		1	
	2-Fluorobiphenyl	48–120		35-125		2	
	2-Fluorophenol	20–120		37–125		3	
	Nitrobenzene-D5	41–120		37–125		4	
	Phenol-D5	20–120		30-125		5	
	Terphenyl-D14	51–135		32–125		6	
	Internal Standards:						
	1,4-Dichlorobenzene-D4					1	
	Naphthalene-D8					2	
	Acenaphthene-D10					3	
	Phenanthrene-D10					4	
	Chrysene-D12					5	
	Perylene-D12					6	

TABLE 4.1
RLs for Method SW8081A

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW-846 8081A	α-BHC	0.05	µg/L	1.7	µg/Kg
Pesticides	β-BHC	0.05	µg/L	1.7	µg/Kg
	δ-BHC	0.05	µg/L	1.7	µg/Kg
	γ-BHC (Lindane)	0.05	µg/L	1.7	µg/Kg
	α-Chlordane	0.05	µg/L	1.7	µg/Kg
	γ-Chlordane	0.05	µg/L	1.7	µg/Kg
	Total Chlordane	0.5	µg/L	30	µg/Kg
	4,4'-DDD	0.1	µg/L	3.3	µg/Kg
	4,4'-DDE	0.1	µg/L	3.3	µg/Kg
	4,4'-DDT	0.1	µg/L	3.3	µg/Kg
	Aldrin	0.05	µg/L	1.7	µg/Kg
	Dieldrin	0.1	µg/L	3.3	µg/Kg
	Endosulfan I	0.05	µg/L	1.7	µg/Kg
	Endosulfan II	0.1	µg/L	3.3	µg/Kg
	Endosulfan Sulfate	0.1	µg/L	3.3	µg/Kg
	Endrin	0.1	µg/L	3.3	µg/Kg
	Endrin Aldehyde	0.1	µg/L	3.3	µg/Kg
	Endrin Ketone	0.1	µg/L	3.3	µg/Kg
	Heptachlor	0.05	µg/L	1.7	µg/Kg
	Heptachlor Epoxide	0.05	µg/L	1.7	µg/Kg
	Methoxychlor	0.5	µg/L	17	µg/Kg
	Toxaphene	5.0	µg/L	170	µg/Kg

TABLE 4.2
QC Acceptance Criteria for Method SW8081A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW-846 8081A	α-BHC	60-128	≤ 30	55-125	≤ 50
Pesticides	β-BHC	46-126	≤ 30	54-127	≤ 50
	δ-BHC	46-136	≤ 30	57-130	≤ 50
	γ-BHC (Lindane)	30-146	≤ 30	59-123	≤ 50
	α-Chlordane	63-123	≤ 30	54-121	≤ 50
	γ-Chlordane	55-120	≤ 30	48-124	≤ 50
	Total Chlordane	50-120	≤ 30	50-120	≤ 50
	4,4'-DDD	50-139	≤ 30	50-139	≤ 50
	4,4'-DDE	48-137	≤ 30	68-126	≤ 50
	4,4'-DDT	47-138	≤ 30	46-135	≤ 50
	Aldrin	42-138	≤ 30	47-120	≤ 50
	Dieldrin	52-129	≤ 30	55-125	≤ 50
	Endosulfan I	45-120	≤ 30	41-147	≤ 50
	Endosulfan II	42-130	≤ 30	37-141	≤ 50
	Endosulfan Sulfate	45-137	≤ 30	55-135	≤ 50
	Endrin	45-134	≤ 30	61-133	≤ 50
	Endrin Aldehyde	48-137	≤ 30	37-147	≤ 50
	Endrin Ketone	56-134	≤ 30	37-147	≤ 50
	Heptachlor	51-128	≤ 30	51-140	≤ 50
	Heptachlor Epoxide	62-131	≤ 30	66-130	≤ 50
	Methoxychlor	56-150	≤ 30	57-143	≤ 50
	Toxaphene	41-126	≤ 30	31-136	≤ 50
	Surrogates:				
	DCBP	32-135		50-132	
	TCMX	33-138		50-124	

TABLE 5.1
RLs for Method SW8082

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW-846 8082	Aroclor-1016	1.0	µg/L	33	µg/Kg
PCBs	Aroclor-1221	2.0	µg/L	67	µg/Kg
	Aroclor -1232	1.0	µg/L	33	µg/Kg
	Aroclor -1242	1.0	µg/L	33	µg/Kg
	Aroclor -1248	1.0	µg/L	33	µg/Kg
	Aroclor –1254	1.0	µg/L	33	µg/Kg
	Aroclor -1260	1.0	µg/L	33	µg/Kg

TABLE 5.2
QC Acceptance Criteria for Method SW8082

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW-846 8082	Aroclor-1016	40-144	≤ 30	41-138	≤ 50
PCBs	Aroclor -1221	41–136	≤ 30	45-136	≤ 50
	Aroclor -1232	41–136	≤ 30	45-136	≤ 50
	Aroclor -1242	39–150	≤ 30	43-150	≤ 50
	Aroclor -1248	41–136	≤ 30	44-136	≤ 50
	Aroclor -1254	29–141	≤ 30	41-141	≤ 50
	Aroclor -1260	45-145	≤ 30	61-131	≤ 50
	1016/1260 Mix	50-135	≤ 30	40-130	≤ 50
	Surrogate:				
	DCBP	32-135		50-132	
	TCMX	33-138		50-124	

TABLE 6.1
RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
SW-846 6010B	Arsenic	15	ug/L	3	mg/Kg
Metals	Beryllium	5	ug/L	1	mg/Kg
	Cadmium	5	ug/L	1	mg/Kg
	Chromium	10	ug/L	2	mg/Kg
	Copper	25	ug/L	5	mg/Kg
	Lead	10	ug/L	2	mg/Kg
	Nickel	40	ug/L	8	mg/Kg
	Selenium	35	ug/L	7	mg/Kg
	Silver	10	ug/L	2	mg/Kg
	Thallium	25	ug/L	5	mg/Kg
	Zinc	60	ug/L	4	mg/Kg

TABLE 6.2
QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW-846 6010B	Arsenic	75-125	≤ 20	75-125	≤ 30
Metals	Beryllium	75-125	≤ 20	75-125	≤ 30
	Cadmium	75-125	≤ 20	75-125	≤ 30
	Chromium	75-125	≤ 20	75-125	≤ 30
	Copper	75-125	≤ 20	75-125	≤ 30
	Lead	75-125	≤ 20	75-125	≤ 30
	Nickel	75-125	≤ 20	75-125	≤ 30
	Selenium	75-125	≤ 20	75-125	≤ 30
	Silver	75-125	≤ 20	75-125	≤ 30
	Thallium	75-125	≤ 20	75-125	≤ 30
	Zinc	75-125	≤ 20	75-125	≤ 30

TABLE 7.1
RLs for Method SW7470A/SW7471A

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
SW7470A (W) SW7471A (S)	Mercury	0.2	µg/L	0.1	mg/Kg

TABLE 7.2
QC Acceptance Criteria for Method SW7470A/SW7471A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7470A/SW7471A	Mercury	75-125	≤ 20	75-125	≤ 30

TABLE 8.1
RLs for Method SW1312/6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
SW-846 1312/ 6010B	Arsenic	*		*	
SPLP Metals	Barium	*		*	
	Cadmium	*		*	
	Chromium	*		*	
	Selenium	*		*	
	Silver	*		*	
		*		*	
SW-846 1312/7470/7471	Mercury	*		*	

*Current laboratory reporting limits should be used.

TABLE 8.2
QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW-846 1312/ 6010B	Arsenic	75-125	≤ 20	75-125	≤ 30
SPLP Metals	Barium	75-125	≤ 20	75-125	≤ 30
	Cadmium	75-125	≤ 20	75-125	≤ 30
	Chromium	75-125	≤ 20	75-125	≤ 30
	Selenium	75-125	≤ 20	75-125	≤ 30
	Silver	75-125	≤ 20	75-125	≤ 30
SW-846 1312/7470/7471	Mercury	75-125	≤ 20	75-125	≤ 30

APPENDIX A

Lab SOP's

Analytical Laboratory SOP Index List
QAPP Addendum, Main Site Evaluation
Former Celotex Site - Chicago, IL

File Name (PDF)	File Description	Pages	Effective Date
0381L_06	Low-Level Sonic Probe Extraction Procedure for the Determination of Semivolatiles in a Solid Matrix	11	1/10/2006
4181_04	Extraction of Chlorinated Herbicides in a Soil Matrix	17	2/14/2006
5711_05	Sample Preparation of Soil, Sediment, Sludge, and Oils for Total Mercury Analysis by Atomic Absorption Cold Vapor Technique	10	5/9/2006
5711_05PA1	Procedural Amendment #1 - Sample Preparation of Soil, Sediment, & Sludge for Total Mercury Analysis by Atomic Absorption Cold Vapor	1	7/26/2006
6006SON_02	Sonic Probe Extraction Procedure for the Determination of Pesticides in a Solid Matrix	10	7/21/2005
02590159_07	Mercury by Cold Vapor Generation	9	3/10/2005
03886119_02	Preparation of Vials for Field Preservation of Soils for Volatile Analysis	11	2/24/2006
09491198_04	Determination of Semivolatile Organic Compounds by Method 8270C	29	12/30/2004
13631224_06	Analysis of Pesticides and Polychlorinated Biphenyls (PCBs) in Solid Samples	23	5/11/2006
15671339_07	Synthetic Precipitation Leaching Procedure (SPLP) Nonvolatile Leachates	18	5/16/2005
18631865_06	Analysis of Chlorinated Herbicides in Soil	18	11/22/2005
23042308-07	Determination of Volatile Target Compounds by Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS) in Soils & Solids by Method 8260B	45	7/14/2005
23042308_07_PA1	Procedural Amendment #1 - Determination of Volatile Target Compounds by Capillary Column Gas	1	4/24/2006
57095708_08	Sample Preparation of Sediments, Sludges, & Soils for Analysis of Metals by Atomic Absorption (5709) or Inductively Coupled Plasma Atomic Emission Spectrometry (5708)	11	8/16/2006
83898390_09	Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035	13	2/28/2006
83898390_09_PA1	Procedural Amendment #1 - Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035	1	2/28/2006
87921339_03	Synthetic Precipitation Leaching Procedure (SPLP) Zero Headspace Leachates	13	11/10/2004
Mcio014_06	Vapor Generation for Cold Vapor Mercury Method Using the Leeman Labs PS200	14	8/31/2006
MCIO016_03	Maintenance for the TJA ICAP^{tm} 61E Trace Analyzer Spectrometer	12	10/13/2004
MCIO018_05	Operation of the Thermo Jarrell Ash ICAP^{tm} 61E Trace Analyzer Spectrometer	13	11/3/2005
SOPIO005_10	Quality Control Procedures for Mercury	20	9/5/2006
SOPIO007_08	Preparation of Standards and Solutions	14	11/30/2005
Sopio007_02	A. Reagents	2	9/16/2004
SOPIO007C_05	Basic Principles: C. ICP Solutions (ICAP^{tm} 61E Trace Analyzer)	25	2/21/2005
SOPIO007D_10	D. ICPMS Solutions	24	7/7/2006
SOPIO007D_10_PA1	Procedural Amendment #1 - ICPMS Solutions	2	9/18/2006
SOPIO007E_07	E. Mercury Solutions	8	3/31/2006
SOPIO0014_18	Quality Control Procedures for ICP	24	9/27/2006
SOPIO33_01	Maintenance for the Perkin Elmer Elan 9000 ICP-MS	10	6/19/2003
SOPIO033_01_PA1	Procedural Amendment #1 - Maintenance for the Perkin Elmer Elan 9000 ICP-MS	1	4/24/2006
SOPIO034_05	Operation of and Analysis with the Perkin-Elmer Elan 9000 ICP-MS	18	4/14/2006
SOPIO035_09	Quality Control Procedures for ICP-MS	19	9/28/2006

Uncontrolled Copy

Low-Level Sonic Probe Extraction Procedure for the Determination of Semivolatiles in a Solid Matrix

Reference:

1. Method 3550B, SW-846. Rev. 2, 12/96.

2. *Chemical Hygiene Plan,* Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
MC-OE-002	Ultrasonic Processor Maintenance and Tuning
MC-OE-003	Semivolatile Extract Cleanup Using Gel Permeation Chromatography
SOP-EX-001	Semivolatile Spiking and Calibration Standards
SOP-OE-001	Glassware Cleaning for Organic Extractions

Scope:

This procedure is applicable for the extraction of priority pollutant and target compounds at low ppm levels from soils or solid wastes. Conditions such as high levels of organic compounds may interfere with normal detection limits.

Basic Principles:

A portion of sample to be analyzed is placed in a beaker. Anhydrous sodium sulfate is added to absorb any water that may be present. Surrogate standards are added to each sample to monitor recovery. See SOP-EX-001. An aliquot of 50% acetone in methylene chloride is then added to the sample. The sample is subjected to sonic disruption to disperse the soil and force solvent contact. The organic compounds present in the soil dissolve in the solvent that is then removed. The sample is extracted two additional times with fresh solvent, the solvent fractions are combined and concentrated to about 1 mL. At this time, it is determined if the sample requires

gel-permeation cleanup (GPC). If needed, the extract is cleaned using GPC and concentrated to 0.5 mL. If GPC is not necessary, the extract is brought to 1.0 mL and bottled. The extract is stored in an amber autosampler vial in the freezer until analysis.

Personnel Training and Qualifications:

All personnel performing these techniques should have performed a solvent concentration quad study that yielded acceptable recoveries for semivolatile LCS compounds. Personnel should spend several days working with an experienced preparation technician who has demonstrated their proficiency of the extraction. Also, several batches of semivolatile samples should be performed under the direct observation of another experienced preparation technician to assure the trainee is capable of independent preparation.

Interferences:

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware is rinsed with solvent before use and a method blank is performed with each batch of sample to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or must be disposed of in the designated containers. These will then be transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) may be disposed of in the normal solid waste collection containers.

Sample Handling:

Samples should be extracted within 14 days of collection. All samples should be stored at 2° to 4°C prior to extractions.

Apparatus and Equipment:

1. Sonic probe apparatus for extracting organic components from a soil matrix – Minimum 300W output – Heat systems, Model W-385 or equivalent

2. Kuderna-Danish assembly with appropriate ampule for concentrating the solvent used during the extraction

3. Steam bath

4. Filter paper – Fisher brand glass fiber filter circles or equivalent

5. Balance – Capable of weighing to 0.01 g

6. Beakers – Stainless steel – Assorted sizes

7. Scoop

8. Pipettes – Class A, assorted sizes

9. Solvent dispenser – Brinkman adjustable or equivalent

10. Wash bottles – Teflon

11. Teflon boiling chips

12. Micro Snyder columns

13. GPC – ABC, Zymark or equivalent

14. Disposable pipettes

15. Autosampler vials – Amber, crimp top

Reagents and Standards:

1. Methylene chloride – Pesticide grade or equivalent

2. Acetone – Semivolatile grade or equivalent

3. Sodium sulfate – Reagent grade or equivalent. Bake at 400°C for 4 hours in a shallow pan prior to use to remove organic contaminants. Store in a glass jar for up to 1 year after baking.

Preparation of Glassware:

See SOP-OE-001.

Procedure:

1. Weigh out 30 ± .04 g of sample into a stainless steel beaker. Record the initial weight to the nearest 0.1-g and any comments about the sample in the extraction log.

   The background, MS, and MSD are performed on three separate aliquots of a field sample.

2. Using a scoop, add at least 60 g of anhydrous powdered sodium sulfate and mix well. Additional sodium sulfate may be added to obtain a free-flowing mixture.

   The blank, LCS and LCSD (if applicable) are prepared using 30 ± .04 g of sodium sulfate weighed into a stainless steel beaker. Record the weight on the extraction log.

3. Using pipettes, add 1.0 mL of BNA surrogate standard into the beaker. Also add 1.0 mL of LCS matrix spiking solution to the matrix spike, matrix spike duplicate, laboratory control sample (LCS), and LCSD if applicable. If the sample requires any special compounds in addition to the priority pollutant semivolatile compounds, 1.0 mL of a 100-ppm spike of this compound is added at this time. If the sample requires Appendix IX analysis, add 1.0 mL of Appendix IX mix in addition to the LCS matrix spiking solution to the matrix spike, matrix spike duplicate, and laboratory control sample at this time. See SOP-EX-001.

   EPA Method Deviation: Acid compounds are added at a concentration of 100 ppm in the matrix spiking and LCS solutions so that the concentrations in the extract are within calibration range.

EPA Method Deviation: Double volumes of surrogates and matrix spiking solutions are not added when a sample requires gel-permeation cleanup. Instead, the extract is concentrated one half the normal final volume to make up for the loss on GPC and maintain the limits of quantitation.

4. Using a solvent dispenser, add 100 mL of 50% acetone in methylene chloride.

5. Set up the sonic probe as described in the manual. See MC-OE-002.

6. Immerse the tip of the sonic probe approximately 1 to 2 cm below the surface of the liquid in the beaker containing the sample and above the sediment layer.

7. Disrupt the sample using a medium tip at full output and a process time/timer of 1:30.

   NOTE: This is equivalent to 3 minutes, 50% duty cycle as described in the EPA method.

8. Remove the probe from the sample and decant the liquid through Fisher glass fiber filter paper into a vacuum flask.

   NOTE: Be sure to turn the vacuum off immediately after solvent is no longer observed dripping from the funnel.

9. Using a solvent dispenser, add 100 mL of fresh solvent to the sample and repeat steps 6 through 8.

10. Using a solvent dispenser, add 100 mL of fresh solvent to the sample and repeat steps 6 through 8. Pour the liquid and solids from the beaker onto the filter paper. Using a wash bottle, rinse the beaker and filter paper with approximately 30 mL of 50% acetone in methylene chloride.

Before placing the probe into another sample, wipe the probe using a paper towel wetted with deionized water to remove any soil present from the previous sample. Rinse the probe with acetone to remove water followed by a methylene chloride rinse.

11. Pour the collected extract into a Kuderna-Danish assembly containing a Teflon boiling chip. Place a 3-ball Snyder column on the set-up, wet the column with methylene chloride, and concentrate over a steam bath which is at 84° to 99°C until the apparent volume in the ampule reaches 1 to 2 mL. Allow the sample to cool 10 minutes. Approximately 3 mL will condense into the ampule during this time.

    This steam bath temperature ensures concentration in a reasonable length of time.

12. Attach the ampule of the K-D to a micro-Snyder column, and concentrate the extract to below 1 mL. Allow the sample to cool.

13. If GPC is not needed, go to step 17. If GPC is needed, dilute the extract to 10 mL with methylene chloride.

14. Place the extract into the appropriate GPC queuing area for storage until the cleanup is performed. See MC-OE-003.

15. Once the GPC clean up is completed, place the extract in a Kuderna-Danish containing a Teflon boiling chip. Place a 3-ball Snyder column on the set-up, wet the column with methylene chloride, and concentrate over a steam bath at 80° to 90°C until the apparent volume reaches 1 to 2 mL. Allow the sample to cool at least 10 minutes. Approximately 3 mL will condense into the ampule at this time.

    This steam bath temperature ensures concentration in a reasonable length of time.

16. Attach the ampule of the K-D to a micro-Snyder column, and concentrate the extract to below 0.5 mL. Allow the sample to cool.

    Method Deviation: The joint of the KD is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

17. Bring to a final volume of 1.0 mL (0.5 mL if GPC was performed) with methylene chloride. The final volume is determined by placing the extract into an amber autosampler vial and comparing the level in the vial to a reference vial containing the exact targeted final volume. Methylene chloride is added to the extract using a disposable pipette until exactly the same level is in both vials. If too much solvent is added to the sample vial, remove the extract from the vial and concentrate it by microsnydering to slightly less than the targeted final volume and rebottle. Cap the vial and store in the freezer until analysis. Record the final volume in the extraction log.

Calculations:

See analysis method.

Statistical Information/Method Performance:

See analysis method.

Quality Assurance/Quality Control:

For each batch of samples extracted, a blank, a laboratory control sample (LCS) (sodium sulfate blank spiked with all compounds to be determined carried through the entire procedure), a matrix spike, and matrix spike duplicate must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead. A batch is defined as the samples to be extracted in any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Revision Log:

Ver. #	Effective Date	Change
00	11/05/96	Previous Issue
01	09/15/97	Major changes are as follows: • Added Holding Times • Adjusted bath temps • Added statement of following specific client and state requirements to Quality Assurance
02	01/29/98	Major changes: • Updated method reference
03	04/05/99	Major changes are as follows: • Update glassware cleaning • Procedure - Clarified final volume determination • Quality Assurance - Batch per day • Reagents - Added length of time Na_2SO_4 can be stored after baking • Incorporated procedural amendment

Ver. #	Effective Date	Change
04	05/15/01	Major changes are as follows: • Cross Reference – Section added • Apparatus – Clarified • Procedure – Clarified/update spiking
05	12/05/03	Major changes are as follows: • QA -- Reformatted to Level 3 • Added Interferences section
06	JAN 1 0 2006	Major changes are as follows: • Incorporated Procedural Amendment #1 • Added reference to *Chemical Hygiene Plan*

0381L_06.DOC
110905

Prepared by: (692) Date: 11-9-05
Senior Chemist, Group Leader

Approved by: Date: 12/21/05
Semi-Volatiles by GC Management

Approved by: Date: 11-25-05
Organic Extraction Management

Approved by: Elaine Stoltzfus Date: 12/27/05
Quality Assurance

Extraction of Chlorinated Herbicides in a Soil Matrix

Reference:

1. Methods 3550B, and 8151A, USEPA SW-846, Third Edition.

2. *Sonicator Ultrasonic Processor and Cell Disruptor Operations Manual*, Sound Heat Systems, Inc., 1985.

3. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
Analysis #1863, 1865, 0344, 5592	Analysis of Chlorinated Herbicides in Soil
MC-OE-002	Ultrasonic Processor Maintenance and Tuning
SOP-OE-001	Glassware Cleaning for Organic Extractions

Scope:

This method is suitable for the extraction of chlorinated herbicides in soils and solid wastes.

Basic Principles:

A portion of sample to be analyzed is placed in an acid rinsed beaker. Acidified sodium sulfate is added to absorb any water that may be present. The mixture is acidified with hydrochloric acid. Surrogate standards are added to each sample to monitor recovery. (See analysis method for preparation.) An aliquot of solvent is then added to the sample. The sample is subjected to sonic disruption to disperse the soil and force solvent contact. The organic compounds present in the soil dissolve in the solvent that is then removed. The sample is extracted two additional times with fresh solvent. The

solvent fractions are filtered into a flask containing acidified sodium sulfate. The filtrate is then poured through a funnel containing glass wool to remove the sodium sulfate and into a Kuderna-Danish assembly. The sample is concentrated on a steam bath, then 37% KOH and deionized water are added and the sample is again concentrated to remove all solvent.

The aqueous solution is transferred to a separatory funnel and extracted with methylene chloride. The solvent is discarded and the aqueous solution is acidified with sulfuric acid, and extracted with ethyl ether. The ether fractions are placed in a flask containing acidified sodium sulfate and left to sit for at least 2 hours. Then the solvent is transferred to a K-D apparatus and concentrated to 10-mL.

Methanol is added and the sample is subjected to esterification. The extract is then concentrated to 2-mL using nitrogen blow down technique. Hexane is added to adjust the final volume and the extract is florisiled.

Personnel Training and Qualifications:

All personnel performing these techniques should have performed a methylation quad study that yielded acceptable recoveries for chlorinated herbicides compounds. Personnel should spend several days working with an experienced preparation technician who has demonstrated their proficiency of the extraction. Also, several batches of herbicide soil samples should be performed under the direct observation of another experienced preparation technician to assure the trainee is capable of independent preparation.

Interferences:

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware is rinsed with solvent before use and a method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Always add acid to water to reduce fuming and bumping. The 25% sulfuric acid and 37% KOH get hot when prepared. Always wear gloves when handling these solutions. In order to reduce the heating of the solutions, the deionized water can be chilled in an ice bath prior to preparation of these reagents.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Always wear gloves when handling the diazald and avoid inhaling diazomethane gas. Both are extremely toxic, severely irritating, and have been cited as carcinogens. See specific safety instructions for this procedure listed in the esterification section.

Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus. All solvent waste generated from this preparation must be collected for recycling (if applicable) or must be disposed of in the designated containers. These will then be transferred to the lab wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) may be disposed of in the normal solid waste collection containers. All waste generated from esterification should be placed in a beaker in the hood and should only be added to the solvent waste stream in the lab-wide disposal facility.

Sample Handling:

Samples should be extracted within 14 days of collection. All samples should be stored at 2° to 4°C prior to extraction.

Apparatus and Equipment:

1. Sonic Probe apparatus (with a minimum of 300W output) for extracting organic components from a soil matrix
2. Kuderna-Danish assembly with appropriate ampule for concentrating the solvent used during concentration
3. Steam bath – VWR/LLI Model #1127 or equivalent
4. Filter paper – Whatman #3 or equivalent
5. N-Evap with nitrogen supply
6. 125-mL separatory funnel
7. pH meter or paper – assorted ranges
8. Glass wool – acid rinsed
9. Peroxide test strips
10. Diazomethane generator
11. Boiling chips – Teflon
12. Vials – assorted sizes
13. Drying columns
14. Beakers – Stainless steel, assorted sizes
15. Pipettes – Class A, assorted sizes

16. Graduated cylinders, Class A, assorted sizes

17. Solvent dispenser – Brinkmann, adjustable

18. Pipettes – disposable

19. Balance – capable of weighing to 0.01 g

20. Wash bottles – Teflon

21. Vials – assorted sizes

22. Volumetric flasks – Class A, assorted sizes

23. Erlenmeyer flasks – assorted sizes

24. Syringes – assorted sizes

25. Micropipetter

26. Powder funnels

27. Glass rods

28. Test tubes

Reagents and Standards:

1. Methylene Chloride – pesticide grade or equivalent

2. Acetone – pesticide grade or equivalent

3. Hexane – pesticide grade or equivalent

4. Ethyl ether, high purity, nonpreserved – Use peroxide test strips to ensure it is free of peroxides.

5. Phosphoric Acid – ACS grade

6. Sulfuric Acid – Instra-Analyzed grade or equivalent

7. Hydrochloric Acid – ACS grade

8. Diazald (N-methyl-N-nitroso-p-toluenesulfonamide)

9. Alcohol GR

10. Potassium hydroxide, (KOH) (37% w/v) – Dissolve 37 g of ACS grade KOH into approximately 80 mL of deionized water in a 100-mL volumetric flask. Shake until the KOH goes into solution. Using a wash bottle, dilute to volume with deionized water. Store at room temperature in a glass bottle. Reagent is stable 1 year. (Equivalent weights and volumes can be used as long as the ratio remains constant).

11. Phosphate buffer (0.1 M) – Dissolve 12 g of sodium phosphate (NaH_2PO_4) in approximately 800 mL of deionized water in a 1000-mL volumetric flask. Shake until the sodium phosphate goes into solution. Using a wash bottle, dilute to volume with deionized water. Add phosphoric acid to adjust the pH to 2.5 ± 0.05 using a pH meter. (Equivalent weights and volumes can be used as long as the ratio remains constant.)

12. Sodium sulfate, acidified, Reagent grade or equivalent – Bake at 400°C for 4 hours prior to use, to remove organic contaminants. Store in a glass jar for up to 1 year after baking. Before use add 1.0 mL of concentrated sulfuric acid to 1 kg of sodium sulfate in a 2-L beaker and slurry with ethyl ether. Remove ethyl ether by placing the mixture on a steam bath. Confirm the mixture is below pH of 4 by adding 1 g of the resulting solid to 5 mL of

deionized water and check pH. Store at 130°C or in a dessicator. Up to 100 g per sample is needed. (Equivalent weights and volumes can be used as long as the ratio remains constant).

13. Sulfuric Acid (10% v/v) – Dilute 100 mL concentrated sulfuric acid (Instra-analyzed grade or equivalent) into approximately 800 mL deionized water in a 1000-mL volumetric flask. Using a wash bottle, dilute to volume with deionized water. Store at room temperature. Reagent is stable 1 year from preparation. (Equivalent volumes can be used as long as the ratio remains constant).

14. Sulfuric Acid (25% v/v) – Dilute 25 mL concentrated sulfuric acid (Instra-analyzed grade or equivalent) into approximately 80 mL deionized water in a 100-mL volumetric flask. Dilute to volume with deionized water. Store at room temperature. Prepare fresh daily. (Equivalent volumes can be used as long as the ratio remains constant).

Preparation of Glassware:

See SOP-OE-001.

Procedure:

1. Using wash bottles, rinse all glassware except 125-mL separatory funnels, beakers, and snyder columns with 10% sulfuric acid solution, followed by deionized water and acetone. This will reduce the amount of alkaline substances present that can react with the organic acids being extracted. Be certain all acetone is completely evaporated before the glassware comes in contact with the samples or low recoveries of Dinoseb will result.

2. Weigh 30 ± .04 grams of sample into a stainless steel beaker. Record the initial weight to the nearest 0.1 g, and any comments about the sample in the extraction log.

The blank, LCS, and LCSD (if applicable) are prepared by weighing 30 ± .04 g of sodium sulfate into a stainless steel beaker. Record the weight on the extraction log.

The background, MS, and MSD are performed on three separate aliquots of a field sample.

3. Add approximately 20 g of acidified sodium sulfate and mix thoroughly. Using a disposable pipette, add concentrated hydrochloric acid to slurry the mixture. Check the pH using narrow range pH paper (1.0 to 2.5). If the pH is not below 2, add more acid until this pH is achieved. Alternatively, 10 mL of phosphate buffer may be added. This will result in a wetter sample, and additional acidified sodium sulfate will be needed.

4. Using pipettes, add surrogate standards and matrix spiking solutions.

   a. Surrogates: Add 1.0 mL Herb Surrogate to all samples, blanks, and spikes.

   b. Spiking Solutions: Spiking solutions are added to the laboratory control sample (LCS), LCSD if applicable, matrix spike and matrix spike duplicate samples. The type of spike is determined by an analysis number. Typically they are as follows:

      (1) Analysis #344, 1863, 1865 — 1.0 mL Herb Spike

      (2) Analysis #5592 — 1.0 mL Hexachlorophene Spike

      NOTE: This may change to accommodate client-specific requirements.

If a sample requires any special compounds in addition to the standard list, an appropriate spike containing the compounds is also added at this time.

5. Using a solvent dispenser, add approximately 100 mL of 50% acetone in methylene chloride.

6. Set up the sonic probe as described in the manual. (See MC-OE-002.)

7. Immerse the tip of the sonic probe approximately 1 to 2 cm below the surface of the liquid in the beaker containing the sample and above the sediment layer.

8. Disrupt the sample using a medium tip at full output of 10 and a process time/timer of 1:30. (This is a total time of 1:30 pulse on and 1:30 pulse off.)

9. Remove the probe from the sample and decant the liquid through Whatman #3 filter paper into a vacuum flask.

10. Using a solvent dispenser, add 100 mL of fresh solvent to the sample and repeat steps 7 through 9.

11. Using a solvent dispenser, add 100 mL of fresh solvent to the sample and repeat steps 7 through 9 once more. Pour the liquid and solids from the beaker onto the filter paper. Rinse the beaker and filter paper with approximately 30 mL of 50% acetone in methylene chloride.

    Before placing the probe into another sample, wipe the probe using a paper towel wetted with deionized water to remove any soil present from the previous sample. Rinse the probe with acetone to remove water.

12. Transfer the filtrate to a K-D apparatus and using a wash bottle, rinse the filter flask with approximately 30-mL of 50% methylene chloride and acetone to complete the transfer.

EPA Method Deviation: The 2-hour wait at this point is not necessary since samples always go through the hydrolysis step. The sample is not centrifuged since the extract is vacuum filtered through filter paper. This eliminates fine particles in the extract.

13. Add a boiling chip to the K-D apparatus and attach a Snyder column, wet the column with methylene chloride and concentrate over a steam bath that is at 85° to 99°C to approximately 5 mL.

    This steam bath temperature ensures concentration in a reasonable length of time.

14. Using a pipette, add 5 mL of 37% KOH solution, and using a graduated cylinder, add 30 mL of deionized water. Also add an additional boiling chip.

15. Reflux on a steam bath at 80° to 85°C (60° to 65°C for all samples from North Carolina) for approximately 30 minutes (1 to 2 hours for samples from North Carolina). All methylene chloride should be evaporated. Allow to cool approximately 10 minutes before transfer to 125-mL separatory funnel, to avoid bubbling in separatory funnel when methylene chloride is added.

    This steam bath temperature ensures concentration in a reasonable length of time.

16. Transfer the aqueous solution to a 125-mL separatory funnel. Using a wash bottle, rinse the K-D with deionized water to complete the transfer.

17. Extract for 2 minutes with 50 mL of methylene chloride. Discard the methylene chloride (lower) layer.

    EPA Method Deviation: This extraction is sufficient to force the herbicide salts into the aqueous solution. Additional extraction with methylene chloride has been found to be unnecessary and results in a loss of hexachlorophene.

18. Using a graduated pipette, acidify the solution to pH <2 with cold 25% sulfuric acid. This requires approximately 5 mL of the acid solution.

19. Perform a 2-minute ethyl ether extraction by shaking the sample with 40 mL of ethyl ether. Drain the aqueous layer into a clean, acid rinsed flask. Place the ethyl ether (top) layer into an acid rinsed flask containing at least 30 grams of acidified sodium sulfate. (Sodium sulfate must be in a quantity so that the sample is completely dried). Return the aqueous layer to the separatory funnel for the next extraction.

20. Perform two additional 1-½ minute ethyl ether extractions by shaking the sample with 20 mL of ethyl ether. Drain the aqueous layer into an acid rinsed flask. Place the ethyl ether layer into the acid rinsed flask that contains 20 grams of acidified sodium sulfate. Return the aqueous layer to the separatory funnel for the next extraction.

21. Allow the extract to remain in contact with the acidified sodium sulfate for a minimum of 2 hours while covered. If the sodium sulfate is not free-flowing, add additional acidified sodium sulfate until all the water is removed. Residual water will hinder the methylation step and therefore must be removed with sodium sulfate before proceeding.

22. Transfer the extract through an acid rinsed funnel containing acid rinsed glass wool into a K-D apparatus. Break up the sodium sulfate with a glass rod. Using a wash bottle, rinse the flask and funnel with two approximately 30-mL aliquots of ethyl ether to complete the transfer.

23. Add a boiling chip to the K-D apparatus and attach a Snyder column, wet the column with ethyl ether and concentrate over a steam bath, which is at 85° to 99°C until the apparent volume in the ampule reaches 1 mL.

    This steam bath temperature ensures concentration in a reasonable length of time.

24. After the sample has cooled, using a syringe, add 0.5 mL of methanol. Using a wash bottle, bring the extract to 10 mL with ethyl ether.

    EPA Method Deviation: Isooctane is not added since the final volume is 10 mL, not 4 mL as written in the method. The isooctane is added to prevent solvent from "blowing off" during methylation. However, since the final volume is greater than the EPA method volume, the isooctane is not needed.

25. Set up diazomethane generator and esterify the extract as described below:

    a. Safety precautions

       Diazald is a carcinogen. Wear gloves at all times during this procedure. Perform esterification in a hood. Avoid inhalation of diazomethane.

       Avoid using etched or scratched glassware and ground glass joints, do not heat over 90°C explosion may result. The generator must be set up in a hood containing no electrical appliances or steam baths. The additional heat and electrical hazard must be avoided.

    b. Diazomethane generator

    c. Procedure

       (1) Prepare the diazomethane solution. Reagents **must** be mixed in the following order: Place 5 grams of KOH in a 125-mL Erlenmeyer flask, using a pipette, add 8 mL of deionized water. Allow the solution to cool, then using a graduated cylinder, add 25 mL of reagent alcohol and using a reagent pump, add 25 mL of ethyl ether. Fill at least two 40-mL vials with ethyl ether for rinsing the generator between samples.

(2) Just prior to starting the procedure, working under the hood, add approximately 3 grams of diazald (5 or 6 pipettefuls) to the KOH solution. Clamp the Erlenmeyer in place. The amount of diazald needed depends on the number of samples extracted. Use 3 g for eight to ten samples. If more than ten samples are extracted, prepare a second diazomethane solution for use during esterification.

(3) Fill a beaker with hot water (80° to 85°C) from the steam bath and hold under the Erlenmeyer containing the diazomethane solution. At the same time, hold a rinse vial of ethyl ether at the end of the generator.

(4) When the rinse vial begins to turn yellow, remove the vial and begin placing samples at the end of the generator. Using a rinse vial, rinse the generator between samples. Be sure each sample turns bright yellow before going on to the next sample. This is to ensure that esterification is complete. If the yellow color does not persist in all of the samples after methylation for the group is complete, remethylate the samples that are no longer yellow.

(5) After esterification, N-Evap the samples to 2 mL. **Do not use heat above 40°C.**

(6) Using a wash bottle, adjust the final volume to 10 mL with hexane.

(7) Florisil the sample as follows:

(a) Prepare a 2-gram florisil cartridge by rinsing two times with 3 mL of hexane. Discard the rinseate.

(b) Using a pipette, add 2 mL of extract to the cartridge. Elute to just above the meniscus at a flow rate of 5 mL per minute. Collect eluant in a test tube.

(c) Place 4 mL of 50% ethyl ether in hexane in a graduated cylinder. Elute the florisil cartridge by slowly adding the solvent mixture to the cartridge. Collect the rinses in the test tube.

(8) Add 100 µL of Herb Internal Standard to each sample, blank, and spike sample. Use a dedicated syringe or micropipettes for this purpose.

(9) N-Evap the extract to just below 2 mL. Using a disposable pipette and a 2-mL volumetric flask, bring to exactly 2 mL with hexane.

(10) Using a disposable pipette, bottle the extract in a clear autosampler vial labeled with the sample number and an "F." Place the remainder of the unflorisiled extract in another 12-mL vial.

(11) If the florisiled extract is colored or has a strong odor, make a dilution of the extract. Bottle in an autosampler vial and a 12-mL vial. Label the vials "F Dfx."

Calculations:

See analysis method.

Statistical Information:

See analysis method.

Quality Assurance/Quality Control:

For each batch of samples extracted, a blank, a laboratory control sample (LCS), (sodium sulfate blank spiked with compounds to be determined carried through the entire procedure) a matrix spike, and a matrix spike duplicate must be extracted. If there is limited sample that prevents the preparation of the MS/MSD then an LCSD must be prepared instead. A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, agency, or state has more stringent QC or batch requirements, these must be followed instead. See the GC analysis method for specifics on compounds in the spiking solution.

Revision Log:

Ver. #	Effective Date	Change
00	02/24/97	Previous Issue
01	12/10/97	Major changes are as follows: • Updated method reference • Changed initial sample weight • Changed names of spiking solutions • Added adjusted bath temps for North Carolina
02	01/06/00	Major changes are as follows: • Basic Principles – Updated location of standards prep info. • Preparation of Glassware – Changed wording. • Quality Assurance – Incorporated Procedural Amendment #1.
03	01/13/04	Major changes are as follows: • Updated document to Level 3 format • Added Interferences section • Updated Apparatus and Equipment section • Clarified Procedure section

Ver. #	Effective Date	Change
04	FEB 14 2006	Major changes are as follows: • Added reference to Chemical Hygiene Plan

4181_04.DOC
010306

Prepared by: _____ (092) Date: 1-3-06
Senior Chemist Group Leader

Approved by: _____ Date: 1-23-06
Organic Extraction Management

Approved by: _____ Date: 1/19/06
Pesticide Residue Analysis Management

Approved by: Elaine Stoltzfus Date: 1/31/06
Quality Assurance

Uncontrolled Copy

Sample Preparation of Soil, Sediment, Sludge, and Oils for Total Mercury Analysis by Atomic Absorption Cold Vapor Technique

Reference:

Method 7471A, *Test Methods for Evaluating Solid Waste*, Revision 1, SW-846, US EPA, September 1994 (**Modified**).

Cross Reference:

Document	Document Title
SOP-IO-001	Preservation, Storage Conditions, and Holding Times for Inorganic Samples
SOP-IO-005	Quality Control Procedures for Mercury
SOP-IO-007	Preparation of Standards and Solutions
SOP-IO-007, Section H	Prep Room Solutions (Solids)
SOP-IO-011	Inorganic Analysis Safety and Waste Handling Procedures
SOP-IO-012	Calculations Used by the Inorganics Group

Purpose:

This digestion procedure is used to prepare soil, sediment, sludge, and oil samples for measurement of mercury by atomic absorption cold vapor technique following SW-846 protocol.

NOTE: For oil samples, weigh 0.2 g of sample into a 4-oz. polypropylene container.

Scope:

This digestion procedure is used by the Metals Department of the Environmental Sciences Division.

Reference Modifications:

To increase efficiency, polypropylene containers are used in place of BOD bottles. Prior to analysis, after excess potassium permanganate is reduced with sodium chloride/hydroxylamine hydrochloride solution, samples are adjusted to 100 mL in volumetric flasks. This allows aliquots to be taken as required for analysis; aliquots cannot be taken when BOD bottles are used. No impact on the quality of the data generated using this modification has been observed.

Basic Principles:

Samples are digested with aqua regia and potassium permanganate to oxidize mercury compounds to mercuric ions and eliminate possible interference from sulfide. Samples high in chlorides require additional permanganate. At the time of analysis, excess permanganate is reduced with sodium chloride/hydroxylamine hydrochloride. Mercuric ions are reduced to mercury metal using stannous chloride. Mercury measurement is performed using mercury cold vapor technique.

Personnel Training and Qualifications:

Training and proof of proficiency for this procedure includes but is not limited to the following:

1. Review and understanding of this procedure
2. Trainee observing trained analyst performing procedure
3. Trainer observing trainee performing procedure
4. Review of trainee's data by trainer
5. Acceptable performance on quad studies for this procedure

6. Documentation of critical steps in training process

Interferences

Not applicable to this procedure.

Safety Precautions and Waste Handling:

Refer to SOP-IO-011.

Sample Handling:

Refer to SOP-IO-011.

Apparatus and Equipment:

1. Polypropylene containers, 4 oz
2. Balance, capable of reading 0.1 mg
3. 100-mL volumetric flasks or other appropriate Class-A volumetric flasks
4. Polypropylene or glass covers

Reagents and Standards:

For reagent preparation, shelf life, and storage conditions, see SOP-IO-007.

1. Hydrochloric acid, HCl, 36.5% to 38.0%, Baker Instra-Analyzed reagent, 1.194 g/mL, or equivalent
2. Nitric acid, HNO_3, 70.0% to 71.0%, Baker Instra-Analyzed reagent, 1.428 g/mL, or equivalent

3. Aqua regia – Mix 30 mL of HCl with 10 mL of HNO_3. Prepare immediately before use. Prepare only enough for daily use.

4. Potassium permanganate, $KMnO_4$, Baker Analyzed reagent, ACS, or equivalent

5. Sodium chloride, NaCl, Fisher, certified ACS, or equivalent

6. Hydroxylamine hydrochloride, $NH_2OH{\cdot}HCl$, Fisher, certified ACS, or equivalent

7. Potassium permanganate solution (5%) – Weigh approximately 50 g of potassium permanganate ($KMnO_4$) into a 600-mL beaker. Transfer the $KMnO_4$ into a 1000-mL volumetric flask using deionized water. Using a stir plate, stir until the $KMnO_4$ is dissolved. Remove the spin bar and dilute to volume with deionized water.

8. Sodium chloride/hydroxylamine hydrochloride solution – Weigh approximately 120 g of sodium chloride (NaCl) into a 400-mL beaker. Transfer, using deionized water, to a 1000-mL volumetric flask. Weigh approximately 120 g of hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) into a 400-mL beaker. Transfer, using deionized water, to the 1000-mL volumetric flask containing the NaCl. Add deionized water and swirl to dissolve solids and dilute to volume with water.

9. Environmental Express HotBlock [block digestor]

Procedure:

For sample preservation, storage conditions, and holding times, see SOP-IO-001.

Turn block digestor on and allow block to reach the Control Point setting that provides 95° ± 1°C sample temperature.[1]

Remove snap closure covers from new 4 oz. polypropylene sample containers with a rapid twisting motion. Save covers for later use.

Weigh three 0.2-g aliquots taken from three different areas (combined 0.60 to 0.65 g to the nearest 0.001 g) of a thoroughly homogenized as-received sample into a 4-oz. polypropylene container (for sample batch spiking procedure see SOP-IO-007H and for sample batch quality control requirements see SOP-IO-005). Add four Corning PYREX 5-mm glass beads to the blank 4-oz polypropylene container. (For oil samples, add only one glass bead to the blank container.) Add about 5 mL deionized water and 5 mL of aqua regia solution. Place sample containers in block digestor and heat approximately 2 minutes. Remove sample containers from block and allow to cool somewhat.

Add 50 mL of deionized water and 15 mL of 5% $KMnO_4$ solution and mix. Add additional portions of 5% $KMnO_4$ solution (in 5-mL increments), if necessary, until the purple color persists for at least 15 minutes. (Add the same amount of $KMnO_4$ solution to entire digestion batch).

Transfer sample containers to block digestor. Place a calibrated thermometer in batch blank container. Put a polypropylene cover on each container. When the thermometer indicates 95° ± 1°C, continue heating for 30 minutes.

Remove sample containers from digestion block and allow to cool. Cap container with snap closure cover.

Prior to analysis, remove cover, add 6 mL of sodium chloride/hydroxylamine hydrochloride solution to reduce excess permanganate. Recap and shake to mix. Add reductant in 6-mL increments until $KMnO_4$ is completely reduced. Remove cover and transfer the solution to a 100-mL volumetric flask, adjust volume to the mark with deionized water, and mix. The sample is now ready for analysis.

[1] **NOTE** that the block temperature is different than the temperature of the liquid being digested.

Turn block digestor on by pressing rocker switch located on the right rear panel. Wait about 8 seconds until controller display indicates current block temperature. PRESS Up Arrow key. The display will show Control Point temperature. Confirm Control Point temperature is set to the block temperature that provides 95° ± 1°C sample temperature; adjust Control Point temperature as required.

Control Point Temperature Adjustment (for control panel yellow buttons) – DO NOT PRESS THE LEFT (SCROLL) KEY. TAP the Up or Down Arrow key; a digit will be highlighted. Holding down the Up or Down Arrow keys can change the highlighted digit to increase or decrease its setting. To change to another highlighted digit, TAP the Up or Down Arrow key. When the desired digit is highlighted, HOLD DOWN the Up or Down Arrow key to change the Control Point setting. After the proper Control Point is set, *no further action is necessary. The display will return to the current temperature in* about 10 seconds.

Control Point Temperature Adjustment (for control panel grey buttons) – PRESS and HOLD * key. The display will show the Set Point Temperature. The digits can be changed to the desired value by pressing the up and down arrows while holding the * key.

Control Point Temperature Adjustment (for control panel RKC SA200 buttonless) – Turn the HotBlock on and wait until the display shows the current block temperature (green digits). To change the set temperature, TAP the SET key once. The far right digit of the set value display (orange digits) will flash, indicating the flashing digit can be changed from 0 to 9 by using the up/down arrow keys. PRESS/TAP the up/down arrow keys to reach the desired value for that digit. Use the SHIFT (<R/S) key to move to the next digit and change it using the up/down keys as desired. Continue until you have reached the desired control point temperature. TAP the SET key TWICE to store the new set

NOTE: Maximum temperature of polypropylene cups [containers] is 130°C.

NOTE: For calibration standards, refer to calibration standard preparation instructions in SOP-IO-007, Sections E.1 to E.3. Digest calibration standards following the same procedure and using the same amounts of all reagents as used for the samples.

NOTE: For samples of liquid consistency, increase weight to 1 g.

Calculations:

Please consult SOP-IO-012 for calculation procedures.

Statistical Information/Method Performance:

Not applicable to this method.

Quality Assurance/Quality Control:

For soil, sediment, and sludge batches, perform a method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample with every digestion batch (20 samples or less).

Revision Log:

Ver. #	Effective Date	Change
00	04/23/96	Previous issue
01	12/31/98	Major changes are as follows: • Title revised for clarification • Reference changed for inclusion of Procedural Amendment #3 (update III changes) • Purpose added for compliance with SOP-LA-033 • Reference Modifications added for compliance with SOP-LA-033 • Basic Principle revised for clarification • Scope revised for compliance with SOP-LA-033 • Personnel Training and Qualifications added for inclusion of Procedural Amendment #2 (compliance with SOP-LA-033) • Procedure revised for inclusion of Procedural Amendment #1 (clarification of 0.6 g sample weighing)
02	11/30/99	Major changes are as follows: • Reference Modifications – polypropylene containers replace Erlenmeyer flasks. • Apparatus and Reagents – polypropylene containers replace Erlenmeyer flasks. Block digestor replaces water bath. • Procedure – polypropylene containers replace Erlenmeyer flasks and block digestor replaces water bath.
03	05/16/01	Major changes are as follows: • Incorporated Procedural Amendment #1 • Cross Reference – Section added • Procedure – Clarified spiking • Added Calculation section

Ver. #	Effective Date	Change
04	04/30/04	Major changes are as follows: • Updated to Level 3 format • Deleted use of SPEX Base Oil 20 in the blank and laboratory control sample (Quality Assurance section)
05	MAY 0 9 2006	Major changes are as follows: • Updated Cross Reference table • Added Sample Handling and Statistical Information/Method Performance sections • Procedure section – Incorporated Procedural Amendment 1

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042506

Prepared by: Debra A. Bryan Date: 4-25-06
Specialist, Group Leader

Approved by: Robert Stoch II Date: 4.25.06
Metals Management

Approved by: Elaine Stoltzfus Date: 4/25/06
Quality Assurance

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Analysis 5711WB
Revision 03 PA #1
Procedure Effective Date: 05/10/06
PA Effective Date: JUL 26 2006
Page 1 of 1

Procedural Amendment #1

Procedure Title: Sample Preparation of Soil, Sediment, and Sludge for Total Mercury Analysis by Atomic Absorption Cold Vapor

Reasons for addition(s) or change(s): Change of polypropylene containers

Samples or project affected: All

List change(s) or addition(s) (specify which section): *Remove reference to 4-oz. in Procedure section; item 2.*

Procedure:

2. Weigh three 0.2-g aliquots taken from three different areas (combined 0.60 to 0.65 g to the nearest 0.001 g) of a thoroughly homogenized as-received sample into a 250-mL Erlenmeyer flask (for sample batch spiking procedure see SOP-IO-007H and for sample batch quality control requirements see SOP-IO-005). Add four Corning PYREX 5-mm glass beads to the blank polypropylene container. (For oil samples, add only one glass bead to the blank container.)

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072506

Prepared by: Debra A. Bryan — Specialist, Group Leader — Date: 7-26-06

Approved by: [signature] — Metals Management — Date: 7.26.06

Approved by: Elaine Stoltzfus — Quality Assurance — Date: 7/26/06

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Sonic Probe Extraction Procedure for the Determination of Pesticides in a Solid Matrix

Reference:

1. Method 3550B, USEPA SW-846, Third Edition.

2. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
Analysis # 1363, 1224, 1225, 1866, 4854, 6000, 6001, 6005	Analysis of Pesticides and Polychlorinated Biphenyls (PCBs) in Solid Samples
MC-OE-002	Ultrasonic Processor Maintenance and Tuning
MC-OE-004	Pesticide Extract Cleanup Using Gel Permeation Chromatography
SOP-OE-001	Glassware Cleaning for Organic Extractions
SOP-OE-004	Cleanup Procedures for Pesticides Organic Extractions
SOP-OE-012	Pesticide Extract Concentration Using a Zymark TurboVap II Concentration Workstation

Scope:

This procedure is applicable for the extraction of organochlorine pesticides from soils, solid wastes, or wipes.

Basic Principles:

A portion of sample to be analyzed is placed in a beaker. Anhydrous sodium sulfate is added to absorb any water that may be present. Surrogate standards are added to each sample to monitor recovery. (See the analytical method for preparation of standards). An aliquot of solvent is then added to the sample. The sample is subjected to sonic disruption to disperse the soil and force solvent contact. The organic

compounds present in the soil dissolve in the solvent, which is then removed. The sample is extracted two additional times with fresh solvent. The fractions are then combined, concentrated, and bottled.

Several cleanup procedures may be required to eliminate matrix interferences before the sample can be analyzed. They include: florisil, copper, and gel-permeation cleanup (GPC).

Personnel Training and Qualifications:

All personnel performing these techniques should have performed a solvent concentration quad study that yielded acceptable recoveries for pesticides SW-846 spike compounds. Personnel should spend several days working with an experienced preparation technician who has demonstrated their proficiency of the extraction.

Also, several batches of pesticide soil samples should be extracted under the direct observation of another experienced preparation technician to assure the trainee is capable of independent preparation.

Interferences:

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware is rinsed with solvent before use and a method blank is performed with each batch of sample to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or must be disposed of in the designated containers. These will then be transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) may be disposed of in the normal solid waste collection containers.

Sample Handling:

Samples should be extracted within 14 days of collection. All samples should be stored at 2° to 4°C prior to extraction.

Apparatus and Equipment:

1. Sonic probe apparatus (with a minimum of 300W output) for extracting organic components from a soil matrix

2. Kuderna-Danish assembly with appropriate ampule for concentrating the solvent used during concentration

3. Steam bath, VWR/LLI Model #1127 or equivalent

4. Filter paper – Whatman #3 or equivalent

5. N-Evap with nitrogen supply

6. Beakers – Stainless steel, assorted sizes

7. Pipettes – Class A, assorted sizes

8. Graduated cylinders – Class A, assorted sizes

9. Solvent dispenser – Beckman adjustable

10. Pipettes – Disposable

11. Balance – Capable of weighing to 0.01 g

12. Teflon wash bottles

13. Vials – Assorted sizes

14. Teflon boiling chips

15. Forceps

16. Scoop

17. TurboVap II concentration workstation w/appropriate concentration tubes– Zymark or equivalent

Reagents and Standards:

1. Acetone – Pesticide grade or equivalent

2. Hexane – Pesticide grade or equivalent

3. Sodium sulfate – Reagent grade or equivalent. Bake at 400°C for 4 hours prior to use to remove organic contaminants. Store in a glass jar for up to 1 year after baking.

4. Methylene chloride – Pesticide grade or equivalent

Preparation of Glassware:

See SOP-OE-001.

Procedure:

1. Weigh out 30 ± .04 g of sample into a stainless steel beaker.

   If the sample is a wipe, remove it from the vial with clean forceps and place in a stainless steel beaker along with any solvent in the vial. Using a wash bottle, rinse the vial with a few mL of acetone/hexane and add the rinseate to the beaker. Record "wipe" in the matrix type section of the extraction log and enter the amount as 1 g in both the extraction log and the extractables database. Also for wipes only, enter the final volume and surrogate/spike volumes in liters in the extractables database. Skip step 2 for all wipe samples. Record the initial weight to the nearest 0.1 g, and any comments about the sample in the extraction log.

   The Blank, LCS, and LCSD (if applicable) are prepared by weighing 30 ± .04 g of sodium sulfate into a stainless beaker. Record the weight on the extraction log.

   The background, MS, and MSD are performed on three separate aliquots of a field sample.

2. Using a scoop, add at least 60 g of anhydrous powdered sodium sulfate and mix well. Additional sodium sulfate may be added to obtain a free-flowing mixture.

3. Using pipettes, add surrogate standards and spiking solutions.

   a. Surrogates – 1.0 mL of SW-846 surrogate is added to all samples, blanks, and spikes.

   b. Spiking solutions – Spiking solutions are added to the laboratory control sample (LCS), LCSD if applicable, matrix spike, and matrix spike duplicate samples. The type of spike is determined by an analysis number. Typically they are as follows:

(1) Analysis #1224, 1225, 1363, 6000, 6001, and 6005 – 1.0 mL SW-846 spike

(2) Analysis #2253 – 1.0 mL captan/captafol spike

(3) Analysis #1866 – 1.0 mL SW-846 spike and an additional LCS/LCSD with 1.0mL Kepone spike

NOTE: This may change to accommodate specific client requirements.

EPA Method Deviation: Double volumes of surrogates and matrix spiking solutions are not added when a sample requires gel-permeation cleanup. Instead, the extract is concentrated one-half the normal final volume after GPC to make up for the loss on GPC and maintain the limits of quantitation.

4. Using a solvent dispenser, add approximately 100 mL of 50% acetone in hexane.

5. Set up the sonic probe as described in the manual. See MC-OE-002.

6. Immerse the tip of the sonic probe approximately 1 to 2 cm below the surface of the liquid in the beaker containing the sample and above the sediment layer.

7. Disrupt the sample using a medium tip at full output of 10 and a process time/timer of 1:30.

   NOTE: This is equivalent to 3 minutes, 50% duty cycle as described in the EPA method.

8. Remove the probe from the sample and decant the liquid through filter paper into a vacuum flask.

NOTE: Be sure to turn the vacuum off immediately after solvent is no longer observed dripping from the funnel.

9. Using a solvent dispenser, add 100 mL of fresh solvent to the sample and repeat steps 6 through 8.

10. Using a Solvent Dispenser, add 100 mL of fresh solvent to the sample and repeat steps 6 through 8 once more. Pour the liquid and solids from the beaker onto the filter paper. Using a wash bottle rinse the beaker and filter paper with approximately 30 mL of 50% acetone in hexane.

    Before placing the probe into another sample, wipe the probe using a paper towel and deionized water to remove any soil present from the previous sample. Rinse the probe with acetone to remove water.

11. Pour the collected extract into a Kuderna-Danish containing a Teflon™ boiling chip. Place a 3-ball Snyder column on the set-up, wet the column with solvent, and concentrate over a steam bath, which is at 85° to 99°C. If the sample requires GPC, skip step 12.

    This steam bath temperature ensures concentration in a reasonable length of time.

12. When the apparent volume in the ampule is 3 to 5 mL, using a graduated cylinder, add approximately 50 mL of hexane directly to the K-D through the Snyder column. Do not allow the ampule to go dry since loss of analytes will result.

    EPA Method Deviation: The K-D is not removed from the steam bath and cooled before adding hexane. The Snyder column is not removed for the solvent addition, therefore, the K-D does not have to be cool.

13. When the apparent volume again reaches 3 to 5 mL, remove the sample from the bath and allow the sample to cool for 10 minutes. Remove the ampule and using a wash bottle adjust the final volume to exactly 10 mL with hexane.

    NOTE: If the sample requires GPC, N-Evap to approximately 1 mL then adjust the final volume to exactly 10 mL with methylene chloride instead of hexane. N-Evap if necessary. Mix thoroughly with a disposable pipette.

    EPA Method Deviation: The Joint of the KD is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

15. If the sample requires GPC, perform GPC cleanup following MC-OE-004. When GPC cleanup is complete, concentrate the extract to final volume 5mL using a Turbovap as described in SOP-OE-012.

16. Florisil the sample 2 mL to 2 mL as described in the Pest Florisil section of SOP-OE-004. Bottle twice in an appropriately labeled crimp-top autosampler vial, and place the remaining extract in an appropriately labeled screw-cap vial. All extracts are stored in the freezer.

17. Copper cleanup may be used to remove sulfur interference from extracts. It is performed when requested by the analytical department. See SOP-OE-004 for this procedure.

Calculations:

See analysis method.

Statistical Information/Method Performance:

See analysis method.

Quality Assurance/Quality Control:

For each batch of samples extracted, a blank, a laboratory control sample (LCS) (sodium sulfate blank spiked with compounds to be determined carried through the entire procedure), a matrix spike, and matrix spike duplicate must be extracted. If there is limited sample that prevents the preparation of the MS/MSD, then LCSD must be prepared instead. A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, agency, or state has more stringent QC or batch requirements, these must be followed. See the GC analysis methods for specifics on compounds in the spiking solutions.

Revision Log:

Ver. #	Effective Date	Change
01	07/30/03	New
02	JUL 21 2005	Major changes are as follows: • Incorporated Procedural Amendments 1 and 2 • Updated Cross References • Updated spiking solutions

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061005

Prepared by: _____ (092) Date: 6-13-05
Senior Chemist/Group Leader

Approved by: _____ Date: 6.27.05
Organic Extraction Management

Approved by: _____ Date: 6/27/05
Pesticide Residue Analysis Management

Approved by: _____ Date: 7/7/05
Quality Assurance

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Mercury by Cold Vapor Generation

Reference:

1. Method 7470A (waters) and 7471A (solids), *Test Methods for Evaluating Solid Waste*, USEPA SW-846, September 1994. **(Modified)**

2. USEPA CLP SOW No. ILM04.0, Exhibit D/Mercury, CLP-M. **(Modified)**

3. Method 245.1, *Methods for Analysis of Water and Wastes*, USEPA 600/4-79-020, Rev. March 1983.

4. Method 245.1, Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA-600/R-94/111, May 1994.

5. USEPA CLP SOW No. ILMO5.2, Exhibit D/Mercury, CLP-M. (Modified)

6. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
Analysis #0494	Sample Preparation of Soil, Sediment, and Sludge for Total Mercury Analysis by Atomic Absorption Cold Vapor Technique
Analysis #0821, 5713, 5714	Sample Preparation of Potable Water and Wastewater for Total Mercury Analysis by Cold Vapor Technique
Analysis #5711	Sample Preparation of Soil, Sediment, Sludge, and Oils for Total Mercury Analysis by Atomic Absorption Cold Vapor Technique
MC-IO-014	Vapor Generation for Cold Vapor Mercury Method Using the Leeman Labs PS200
SOP-IO-001	Preservation, Storage Conditions, and Holding Times for Inorganic Samples
SOP-IO-005	Quality Control Procedures for Mercury
SOP-IO-007	Preparation of Standards and Solutions
SOP-IO-011	Inorganic Analysis Safety and Waste Handling Procedures
SOP-IO-012	Calculations Used by the Inorganics Group

Purpose:

The purpose of this procedure is to detail the instrument parameters used to analyze mercury by cold vapor generation.

Scope:

This procedure is applicable to the determination of mercury in waters, wastewaters, and leachates (#0259) and soils (#0159).

Background Information:

The optimum concentration range for this method is 0.2 to 5.0 ppb. The instrument detection limit for the method using the Leeman Labs PS200II is 0.01 ppb. The following limits of quantitation are used in accordance with the requirements of the governing regulatory agency.

Limits of Quantitation:

	CRDL	Reference Method
		CLP SOW No. ILMO4.0 245.1 CLP-M
CLP WW	0.0002 mg/L	CLP SOW No. ILMO5.2, CLP-M
		CLP SOW No. ILMO4.0 245.5 CLP-M
CLP SW	0.1 mg/kg	CLP SOW No. ILMO5.2, CLP-M

	LOQ	Reference Method
Waters	0.0002 mg/L	EPA SW-846, Method 7470A
Solids	0.1 mg/kg	EPA SW-846, Method 7471A
PW/EW	0.0002 mg/L	EPA 600, Method 245.1
NPDES	0.0002 mg/L	EPA 600, Method 245.1

Reference Modifications:

SW-846 Methods 7470A and 7471A are manual procedures. This SOP is for an automated determination. The chemistries used to do the mercury determinations are the same. No impact on the quality of the data generated using this modification has been observed.

Basic Principles:

The reaction for the mercury analysis is a simple reduction reaction. The mercury (Hg^{++}) is reduced with stannous chloride (Sn^{++}) to liberate mercury metal and Sn^{+4}. The sample is made acidic with hydrochloric acid to maintain the reducing environment of the reaction. Air or inert gas is used to sweep the volatile mercury into the absorption cell in the optical path of the atomic absorption spectrophotometer.

Personnel Training and Qualifications:

1. Review and understanding of this procedure.
2. Trainee observing trained analyst performing the procedure.
3. Trainer observing trainee performing the procedure.
4. Review of trainee's data by trainer.
5. Acceptable performance on quad studies for this or equivalent procedure.
6. Documentation of critical steps in the training process.

Reagents and Standards:

(Use the following or equivalent):

1. Nitric acid, 70.0% to 71.0% HNO_3, Baker Instra-Analyzed reagent, 1.428 g/mL; store at room temperature

2. Sulfuric acid, 95.0% to 98.0%, H_2SO_4, 36 N, Fisher reagent, ACS, 1.84 g/mL; store at room temperature

3. Potassium permanganate, $KMnO_4$, Baker Analyzed reagent, ACS

4. Potassium persulfate, 5% $K_2S_2O_8$ Baker Instra-Analyzed reagent, ACS

5. Sodium chloride, NaCl, Fisher, Certified ACS

6. Hydroxylamine hydrochloride, $NH_2OH{\cdot}HCl$, Fisher, Certified ACS

7. Deionized water, Type 2 or better

8. Stannous chloride solution, 10% SnCl, Baker Analyzed reagent, ACS

9. Hydrochloric acid, HCl, 36.5% to 38.0%, Baker Instra-Analyzed reagent, 1.194 g/mL or equivalent

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Refer to SOP-IO-011.

Procedure:

Consult SOP-IO-001 regarding sample preservation, holding times, and storage conditions. Consult Analysis #0821, 5713, 5714, 5711, and 0494 for digestion procedures.

1. Preparation of standard solutions

   For detailed procedures on preparation of standard solutions consult SOP-IO-007, Sections E, G, and H.

2. Instrument setup

   For detailed procedures of the Leeman Labs PS200II automated mercury analyzer, please refer to MC-IO-014.

3. Program parameters

   Leeman Labs PS200II

   Mercury analyzed using the Leeman Labs PS200II – The instrument parameters are preset for the program that was developed for mercury. The parameters for this program are as follows:

	Hg
Instrument mode	Intensity
Calibration mode	Concentration
Sample introduction	Automated
Integration time (sec)	10
Replicates	1

Calibration Standards (mg/L)	Controls	(mg/L)
0.0002	ICV (all other methods)	0.0025
0.0005	ICV (CLP ILM05.2)	0.0020
0.001	CCV	0.001
0.0025	CRA	0.0002
0.005		

NOTE: CCV CONCENTRATION SHOULD BE LESS THAN OR EQUAL THE MAXIMUM CONTAMINATION LEVEL OF 0.002 mg/L FOR POTABLE (PW) AND DRINKING (EW) WATERS.

NOTE: The ICV for CLP ILM05.2 is 0.0020 mg/L.

NOTE: The concentration values and instrument conditions are for Leeman Labs PS200II. All changes from the standard analytical program will be documented on the raw data.

Calculations:

Consult SOP-IO-012 for calculation procedures.

Quality Assurance/Quality Control:

Consult SOP-IO-005 for specific QC protocol and procedures.

Revision Log:

Ver. #	Effective Date	Change
00	01/18/96	Previous Issue
01	05/06/99	Major changes are as follows: • Removed reference note to add a Reference Modification section • Changed Background Information, Procedure, and Quality Assurance section to remove references to old instrumentation • Added reagents to Reagents section • Added Personnel Training and Qualifications section
02	01/03/01	Major changes are as follows: • Added and updated method references.
03	03/26/01	Major changes are as follows: • Added Cross Reference section. • Program Parameters – Changed ICV level to 2.5 • Deleted reference to PS200 instrument throughout the procedure. • Incorporated Procedural Amendment #1
04	05/16/01	Major changes are as follows: • Updated Cross References • Procedure – Updated Procedure 1.
05	07/18/01	Major changes are as follows: • Limits of Quantitation – Changed all units to mg/L Procedure – 3. Changed all units to mg/L

Ver. #	Effective Date	Change
06	03/17/03	Major changes are as follows: • Updated procedure due to change in internal documentation system. • Updated reference section • Updated Limits of Quantitation section
07	MAR 1 0 2005	Major changes are as follows: • Incorporated Procedural Amendment #1 and #2 • Updated Reference section

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Prepared by: Jennifer L. Moyer Date: 02/16/05
Chemist Coordinator

Approved by: Robert Strachan 8H Date: 2.17.05
Metals Management

Approved by: Dorothy M Love Date: 2/24/05
Quality Assurance

Uncontrolled Copy

Preparation of Vials for Field Preservation of Soils for Volatile Analysis

Reference:

1. Method for the Determination of Volatile Petroleum Hydrocarbons (VPH), MA DEP, January 1998.

2. Method for the Determination of Volatile Petroleum Hydrocarbons (VPH), MA DEP, May 2004.

3. EPA Method 5035, Revision 0, SW-846, U.S. EPA, December 1996.

4. EPA Method 5035A, Draft Revision 1, SW-846, U.S. EPA, July 2002.

5. Method for the Field Extraction/Preservation of Soil Samples with Methanol for Volatile Organic Compounds, New Jersey DEP, February 1997.

6. Method AK101 -- For the Determination of Gasoline Range Organics Version 4/8/02.

7. Instructions for EPA Reference Method 25D- Interlaboratory Comparison, Research Triangle Institute, October 1991

8. Chemical Hygiene Plan, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
LOM-SOP-LAB-208	Laboratory Balances
LOM-SOP-LAB-220	Laboratory Notebocks, Logbooks, and Documentation
SOP-SS-017	Preservation and Bottles Room Preservative Traceability
SOP-SS-018	Pipette Dispenser Calibration Procedure
Form 4580	Field Preserved Vial Preparation for Volatile Soils

Scope:

This procedure will cover the preparation of pre-preserved containers to be used in the field for soil sampling of volatile analyses.

Basic Principles:

An aliquot of preservative is placed in a volatile-free container and weighed. The weight of the container and preservative is then captured using the Volatile Preparation program in Parallax. Upon request, the container is sent to the client for use in field preservation of a solid sample. When the container and the soil sample is returned to the lab, it is re-weighed and the actual sample weight is calculated.

Personnel Training and Qualifications:

The initial training consists of observing the procedure being carried out by an experienced analyst allowing for questions and feedback. Following the initial training, experienced analysts are available as a resource until no longer required. Analysts are considered proficient when the procedure can be carried out independently.

Interferences:

Sample contamination could occur if the vial preparation is not done in a volatile free environment, therefore this process must be performed in one of the designated volatile free laboratories. Samples can also become contaminated by diffusion of volatiles through the sample vial septum. A trip blank carried through sampling, storage and handling can act as a check of such contamination.

Safety Precautions:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Methanol is flammable. Containers of this solvent must be kept away from any sources of open flames or sparks. Vials containing methanol must be stored in explosion proof refrigerators.

Sample Handling:

Sample containers must be refrigerated at 2° to 4°C after preparation and should not be kept on the shelf for more than 2 weeks before being discarded or sent into the field for use in sample collection. Packaging of sample containers must follow all DOT regulations.

Apparatus and Equipment:

1. 40-mL vials with Teflon lined septa and screw caps

2. 40-mL vials with stir bars, Teflon lined septa, and screw caps. SciSpec Catalog #376740-MB or equivalent.

3. 125-mL amber glass wide mouth jar with Teflon lined septa and screw caps.

4. Repipette capable of dispensing up to 25 ± 0.25 mL. Refer to SOP-SS-018.

5. 50-µL syringe

6. 1000-mL volumetric flask, class A

7. Analytical balance capable of weighing 0.01 g. Refer to LOM-SOP-LAB-208

8. Label Printer/labels

Reagents and Standards:

1. Methanol – Purge and trap grade, store at room temperature and re-analyze yearly. The methanol used must have been previously tested and approved for use by the labs. See SOP-SS-017 for further information.

2. 8260A/B Surrogate Mix, Restek Catalog #30340 (2500 µg/mL) or equivalent. Store at –10° to –20°C. This standard may be used as is or may be diluted in methanol to a final concentration of 2.5 µg/mL. This standard is used for the GC/MS analyses.

3. Custom a,a,a, trifluorotoluene (TFT), Restek Catalog #54357 (15,000 µg/mL) or equivalent. This standard may be used as is or may be diluted in methanol to a final concentration of 750 µg/L. Store at –10° to –20°C for up to 1 month. This standard is used for the GC analyses.

4. Sodium hydrogen sulfate anhydrous powder, Fluka, Catalog #2316657 or equivalent. Store at room temperature and re-analyze yearly. If compounds are detected above the method detection limit (MDL), prepare another vial and repeat the analysis. If compounds are still detected above the MDL, a new container must be tested and used.

5. Sodium Bisulfate Solution – prepared by diluting 200 ± 5 g of the Sodium *hydrogen sulfate anhydrous into 1000 mL of deionized water in 1000-mL* volumetric flask. Cap and invert at least 3 times to mix. Store at room temperature and re-analyze every 6 months if supply remains. If compounds are detected above the method detection limit (MDL), repeat the analysis. If compounds are still detected above the MDL, remake the solution and test before using.

6. *Deionized* Water – ASTM Type II (water from our in-house deionized system is acceptable)

7. Polyethylene glycol (PEG)- Average molecular weight 400 amu, EM Science preferred. Any lot/vendor must meet a cleanliness level of < 50 mg/kg volatile content.

Procedure:

NOTE: The Parallax VOA Prep application integrates a PC with an analytical balance to collect data directly from the balance. It organizes the data, performs calculations, and stores final results in the Laboratory Information Management System.

The VOA Prep application should be used whenever possible for this procedure to facilitate data transfers and other tracking. However, data may still be recorded traditionally in a logbook. Refer to LOM-SOP-LAB-220.

A. Preparing preservative containers

1. Check to make sure the repipette calibration has been performed.

2. Using the repipette, add the appropriate amount of preservative (see Form 4580) to a clean container. A 40-mL vial is used unless otherwise indicated on Form 4580. For 6119, 6172, and 1169 the surrogate solution

must be added at this time. This can be accomplished by using the repipette as noted above and using the appropriate diluted surrogate solution. Alternatively for 1169 and 6172, the ampulated 8260A/B surrogate standard can be used to add the surrogate to the methanol in the 40-mL vial using a syringe. Add 1 µL of 8260A/B surrogate for every 1 mL of methanol in the vial.

3. Seal the container with a screw cap and septum seal.

4. Label the container with the tracking number.

5. Check to make sure the balance has been calibrated each day before use. Place the container on a zeroed balance and capture the weight electronically to the nearest 0.01 g using the VOA Prep application or record manually in the appropriate logbook.

6. Store the prepared containers on the designated shelf in the refrigerator in the Volatile Prep room.

7. Pull vials as requested via e-mail by client services and place on the designated shelf in the bottles room storage area. The container is now ready to be sent into the field for sample collection.

8. Send a reply e-mail to the client service representative to notify them that the order has been completed.

B. Re-weigh preservative containers after return from the field

1. Using the Volatile Prep application, scan the tracking number on the container. This will bring up the information documented from the preparation step described above. If manual documentation is being

performed, locate the page listing the tracking number in the appropriate logbook.

2. Check to make sure the balance has been calibrated each day before use. Place the container on a zeroed balance and capture the second weight electronically using the VOA Prep application or record manually in the appropriate logbook.

3. The Volatile Prep application will then calculate the net weight. If manually documenting, calculate the net weight (weight of vial, solution, and soil minus the weight of the vial and solution) and document in the net weight column for the appropriate sample.

4. If there are any holding time issues or if weights are outside of the Action Requirement listed on Form 4580, the VOA Prep application automatically sends an e-mail to client services. If manually documenting, fill out a problem form and deliver to the client service representative.

5. Record any unusual observations about the sample in the comment section.

C. Deliver samples to the labs

Once the sample containers have been re-weighed, they must be transported to the laboratory that will be analyzing them. Each department has a designated drop-off spot that is refrigerated. A copy of the associated data print out must accompany the containers to the department.

Calculations:

Calculation of sample weight: Wn = Ws - Wf

Where:

Wf = weight of container + solution (first weight)

Ws = weight of container + solution + soil (second weight)

Wn = net weight of soil sample

Statistical Information/Method Performance:

Not applicable to this procedure

Quality Assurance/Quality Control:

The number of containers requested from client services should include the appropriate amount of extra bottles to serve as QC for the volatile analyses requested.

Revision Log:

Ver. #	Effective Date	Change
00		Previous issue:
	09/10/03	Analysis #0388
	05/30/03	Analysis #0405
	02/18/05	Analysis #1169
01	12/03/05	Major changes are as follows: • Combined the content of the three analyses noted above plus added 2392, 6119, 6169, 6171, 6172, 6173, 6176, 6645, 6647, and 7579

Ver. #	Effective Date	Change
02	FEB 2 4 2006	Major changes are as follows: • Analysis #0069 added • Polyethylene glycol (PEG) added to Reagents and Standards section

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Prepared by: _____ Date: 2/24/06
Specialist, Group Leader

Approved by: _____ Date: 2-24-06
Sample Support Management

Approved by: _____ Date: 2/24/06
Quality Assurance

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

Field Preserved Vial Preparation for Volatile Soils

Dept 25 Soil Prep Options	Analysis	Bottle Code	Preservative Type	Volume of Solution	Weight Requirement	Action Required
GC - Field Preserved MeOH	6169	64	MeOH	10 ml	10 +/- 1	> 15 < 5
GC – Field Preserved MeOH	6647	64	MeOH	5 ml	5 +/- .5	> 7.5 < 2.5
GC - Field Preserved (MA-VPH)	0388	64	MeOH	15 ml	15 +/- 3.75	> 18.75 < 11.25
GC - Field Preserved (AK-101)	6119 *	48	MeOH w/ Surrogate	25 ml	25 +/- 2.5	> 37.5 < 12.5

* Use a 125 mL container.

Dept 21 High Level Soil	Analysis	Bottle Code	Preservative Type	Volume of Solution	Weight Requirement	Action Required
GC/MS - Field Preserved MeOH	6171	14	MeOH	5 ml	5 +/- .5	> 7.5 < 2.5
GC/MS - Field Preserved MeOH	0405	14	MeOH	10 ml	10 +/- 1	> 15 < 5
GC/MS - Field Preserved MeOH	6645	14	MeOH	15 ml	15 +/- 1.5	> 16.5 < 13.5
GC/MS - Field Preserved MeOH (DE)	6172	69	MeOH w/ Surrogate	10 ml	10 +/- 1	> 15 < 5
GC/MS - Field Preserved (NJ)	1169	69	MeOH w/ Surrogate	25 ml	10 +/- 2	< 8 > 12
GC/MS - Field Preserved (Alaska)	6173 *	66	MeOH	25 ml	25 +/- 2.5	> 37.5 < 12.5

* Use a 125 mL container.

Dept 21 Low Level/High Level Combination	Analysis	Bottle Code	Preservative Type	Volume of Solution	Weight Requirement	Action Required
GC/MS - LL Field Preserved NaHS04	2392 **	2 x 93	Sodium Bisulfate	5 ml	5 +/- .5	> 7.5 < 2.5
GC/MS - Field Preserved MeOH (linked to 2392)	7579	14	MeOH	5 ml	5 +/- .5	> 7.5 < 2.5
GC/MS - LL Water Prep	6176 **	2 x 50	DI Water	5 ml	5 +/- .5	> 7.5 < 2.5
GC/MS - Field Preserved MeOH (linked to 6176)	7579	14	MeOH	5 ml	5 +/- .5	> 7.5 < 2.5

** Use a 40 mL vial with a stir bar and low bleed septa.

Dept. 32 Prep Options	Analysis	Bottle Code	Preservative Type	Volume of Solution	Weight Requirement	Action Required
PEG Prep	0069	92	PEG	30 mls	N/A	N/A

4580.02

Basic Principles:

A 1-microliter mixture of organic compounds in methylene chloride is injected onto a fused silica capillary column coated with a relatively non- polar stationary phase, which is enclosed in a temperature controlled oven. A carrier gas, ultra pure helium, passes continuously through the column. The GC oven is temperature programmed and the organic mixture separates into its individual components as it moves along the length of the column. This separation is a function of the polarity and boiling point of the individual compounds. The column empties into a mass selective detector. When a compound reaches the detector, it is bombarded by high energy electrons (70 eV). This causes the compounds to fragment, forming ions. By applying various voltages to lenses in the area where the ions are formed, the positive ions are thrust into a quadruple mass analyzer, which selects for a given mass fragment at a given time. These selected fragments reach an electron multiplier, which detects and generates a signal for each mass fragment. The signals are amplified and sent to a computer making storage and manipulation of the data possible. Target compounds are identified on the basis of relative retention times and spectral match to standards. Standards are injected every 12 hours on each system used for analysis.

Quantification is achieved via use of the internal standard calibration technique. The average relative response factor of a multi-point calibration is used for quantification when the appropriate criteria are met.

Personnel Training and Qualifications:

Education Requirement: Degree in science or relevant experience

Each new chemist will train with an experienced chemist for the first 12 weeks. The first 12 weeks are spent working one-on-one with the trainer. This time may be less if the new chemist has prior experience in the GC/MS Semivolatiles area or relevant analytical chemistry background. Each new chemist receives a training manual outlining the basics of operating the GC/MS and data work up.

During the training period, the new chemist will learn daily maintenance, column and source changing procedures, calibration techniques, data and library search review, and forms generation. They are also required to read all relevant SOPs and EPA methods.

To measure the proficiency of each chemist, several checks have been established. The first is the ability to calibrate for each method. The chemist will run a series of at least five calibration standards and perform the calibration routine. A departmental data validator will then review the curve. They will confirm that relative retention times (RRT) and response factors (RF) match throughout the calibration and ID list. Secondly, each analyst must perform a quad study. This will consist of serial dilutions on a known concentration mixture and analyzing four back-to-back replicates of these dilutions. This process will measure accuracy in dilution preparation as well as reproducibility of results. It is a requirement that quad studies are performed by each analyst on an annual basis.

Interferences:

Method interferences may be caused by impurities in solvents, reagents, and glassware, or other hardware using in the processing of samples. All glassware is solvent rinsed before use and a method blank is performed with each extraction batch to demonstrate that the extraction system is free of contamination.

Safety Precautions and Waste Handling:

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound and reagent should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means are available such as fume hoods, safety glasses, lab coats, and gloves . Refer to the *Lancaster Laboratories Chemical Hygiene Plan* for specific details.

All solvent waste generated from this analysis must be collected for recycling (if applicable) or must be disposed of in designated containers. These will then be

transferred to a lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) must be disposed of in the normal solid waste collection containers or sharps containers, as applicable.

Sample Preservation and Holding Time:

Water samples may be preserved with sodium thiosulfate ($Na_2S_2O_3$) and must be prepared within 7 days of the date collected. Soil samples are not preserved and must be prepared within 14 days of the date collected. Extracts must be analyzed within 40 days of the date extracted.

Extracts must be refrigerated at 2° to 4°C in amber vials.

Apparatus and Equipment:

1. 25-µL syringe

2. Hewlett-Packard Model 5890 (Series I and II) or Hewlett-Packard/Agilent 6890 Gas Chromatograph or equivalent

3. Hewlett-Packard Models 5971, 5972, and Hewlett-Packard/Agilent 5973 Mass Selective Detector or equivalent

4. Thru-Put Systems Target Acquisition Software/Oracle Database or equivalent

Reagents and Standards:

1. 50 ng/µL Solution of decafluorotriphenylphosphine (DFTPP) containing pentachlorophenol, benzidine and DDT, prepared from Absolute Standards, Inc., part # 43030 in methylene chloride or equivalent. Store at 0° - 4°C for up to 6 months.

2. Methylene chloride, pesticide grade. Store at room temperature.

3. Supelco Equity Semivolatile Internal Standard Mix, part # 46955-U or equivalent, 2000 µg/mL in methylene chloride. Ampulated solutions are maintained at <10°C until consumed or manufacturer determined expiration date. Working solution is maintained at room temperature and is replenished daily from ampulated solutions.

4. Calibration Standards – Refer to SOP-EX-001

Procedure:

Refer to the GC/MS Semivolatiles training manual for the specific information on acquiring and processing data.

Internal standard mix is added to all standards and subsequent samples at a concentration of 40 µg/mL. Using a 25-µL syringe, 20 µL of Supelco Equity Semivolatile Internal Standard Mix or equivalent, 2000 ug/mL in methylene chloride are added to the 1 mL of standard or sample extract.

A. Standard preparation – These solutions are used to standardize the GC/MS system every 12 hours and are prepared aproximately every week to 10 days or more frequently if needed based on consumption. See SOP-EX-001 for standard preparation. Calibration standard solutions may be used up to the labeled expiration date or until component degradation is observed.

B. Daily maintenance – Refer to MC-EX-001 for this procedure.

C. Instrument Conditions

Equip a GC/MS (such as referenced under Apparatus and Equipment) in one of the two following manners:

For a 5890/5971 or 5972 and 6890/5973

1. Column – 30M x 0.25 mm ID, 1.0 um df, J&W Scientific DB-5MS or equivalent

2. Injector – Split/splitless operated in splitless mode

3. Injector Temp – 275°C

4. Detector Temp – 300°C

5. Gas – Helium at approximately 1.5 mL/min, constant flow mode

6. Oven Temp – 45°C for 3 minutes, ramp at 8°C/minute to 225°C, then ramp at 12°C/minute to 300°C and hold for 7.5 minutes.

For a 6890/5973

1. Column – 20M x0.18 mm ID, 0.18 um df, J&W Scientific DB-5MS or equivalent

2. Injector – Split/splitless operated in split mode, 30:1 split

3. Injector Temp – 275 °C

4. Detector Temp – 280 °C

5. Gas – Helium at approximately 1.0 ml/min, constant flow mode

6. Oven Temp – 40 °C for 1 minute, ramp at 25 °C/minute to 100 °C, then ramp at 30 °C/minute to 280 °C, followed by another ramp at 25 °C/minute to 320 °C, hold for 2 minutes.

Note: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions that give the performance required by the method are acceptable.

D. Tuning

The GC/MS must be tuned using a 50 ng/µL solution of DFTPP containing pentachlorophenol, benzidine, and DDT.

Frequency	Acceptance Criteria	Corrective Action
Every 12 hours	1. Criteria in Table I 2. DDT breakdown ≤20%* 3. Tailing factors: • Benzidine ≤3 • Pentachlorophenol ≤5	1. Retune. Analysis cannot proceed until tune meets criteria. 2. More aggressive injection port maintenance. 3. Clean the source. 4. Change the column.

***Note:** DDT breakdown greater than 20 percent may be acceptable if you are calibrating for polynuclear aromatic hydrocarbon compounds only. Consult supervisor when this situation occurs.

1. Use only the background-subtracted spectrum of the following when evaluating the DFTPP:

   a. The apex of the scan

   b. The apex of the scan -1

   c. The apex of the scan +1

   d. A three scan average of the above three scans

   e. A five scan average

NOTE: All standards, samples, and associated quality control samples with a particular tune must use the identical conditions of the mass spectrometer.

2. Calculation of DDT breakdown

$$\%\ DDT\ Breakdown = \frac{DDE\ TIC\ AREA + DDD\ TIC\ AREA}{DDE\ TIC\ AREA + DDD\ TIC\ AREA + DDT\ TIC\ AREA} \times 100$$

Where:

DDE and DDD = The breakdown products of DDT

TIC = Total Ion Chromatogram

E. Initial Calibration

Standardization is performed by analyzing at least six levels of calibration standards ranging from 5 µg/mL to 120 µg/mL. (Refer to SOP-EX-001 for the preparation of calibration standards.) Using the internal standard calibration technique an average relative response factor is generated for each compound. Table 3 lists the six internal standards used for the method and the target compounds that are associated with each internal standard. Refer to the GC/MS Semivolatile Training Manual for more specific information. A method detection limit (MDL) standard must be analyzed with each initial calibration. This standard is prepared at the departmental MDL and is not to be included in the calibration curve. All compounds must be detected in the MDL standard. An initial calibration verification (ICV) standard is also to be analyzed with each initial calibration.

Frequency	Acceptance Criteria	Corrective Action
Initially and then when CCCs and/or SPCCs in the daily calibration standard fail criteria. Initially establish with at least six levels of standards and an MDL standard. See Table 2 for a list of the SPCC and CCC compounds.	1. Ave RRF for each SPCC ≥0.05. 2. %RSD for each CCC ≤30%. 3. %RSD for non-CCCs ≤50%. * 4. All compounds of interest must be detected in the MDL standard. 5. The relative retention times of the target compounds must agree within 0.06 relative retention time units. The exception would be in the case of system maintenance. 6. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.	1. Any target analyte with an %RSD of ≤15% should use the average RRF. For any analyte in which the %RSD >15%, use a first degree (linear) fit should be used if the correlation coefficient is ≥0.99. If the CC of the linear fit is <0.99, then a second order (quadratic) fit may be used provided the coefficient of determination is ≥0.99. If both the CC for the linear fit and the CD for the quadratic fit are ≥0.99 for any given analyte, then use the fit with the smallest negative y-intercept. When using a quadratic fit, if the y-intercept quantifies to be greater than the MDL, consult your supervisor immediately or recalibrate. See below for corrective action if the coefficient of determination (COD) for a quadratic fit is <0.99.** 2. If a compound is not detected in the MDL standard, then report to the level of the lowest standard detected. All compounds manually integrated in this standard must be checked for in each sample analyzed under this initial calibration.* 3. More aggressive system maintenance, and recalibrate.

*If these situations occur, your supervisor is to be consulted immediately.

** See USEPA Method 8000B for the calculations associated with non-linear fit types.

With supervisory approval, the following problematic compounds can be allowed to fail the 0.99 coefficient of determination criteria for a quadratic fit:

1,4-Phenylenediamine
4-Aminobiphenyl
3,3'-Dimethylberizidine
4,4'-Methylenebis(2-chloroaniline)
4-Nitroquinoline-1-oxide
1,4-Naphthoquinone
methapyrilene

If the CD is less than 0.99 for any other compound, the system should be inspected for problems and recalibrated. Supervisory approval is required for exceptions to these guidelines.

F. Continuing calibrations

Frequency	Acceptance Criteria	Corrective Action
1. Every 12 hours. 2. Check standard should be run at alternating concentration levels starting with the standard that is at or near the mid-point of the calibration. The check standard run in the next 12 hour tune should be the concentration level above the mid-point followed by the concnetration level below the mid-point in the subsequent 12 hour tune. The concentration of the check standard will continue to be alternated until a new initial calibration is required, at which point the alternating process starts anew.	1. RRF for each SPCC ≥0.05. 2. %Drift for each CCC ≤20%. 3. %Drift for all non-CCCs ≤50%. * 4. The relative retention times of the target compounds must agree within 0.06 relative retention time units. The exception would be for the case of system maintenance. 5. The EICP area for each internal standard must fall within the window of -50% to +100% from average of the areas produced during the last initial calibration. 6. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.	1. If the CCC or SPCC compounds do not meet criteria but all compounds of interest have a %Drift ≤20%, the calibration may be used. ** 2. More aggressive system maintenance or recalibrate

*If these situations occur, your supervisor is to be consulted immediately

**Notification to the data user will occur in the case narrative that is submitted with the data package. *Your supervisor must be consulted.*

In the event that two consecutive continuing calibration check standards fail for the list of target analytes being quantified, then after the appropriate system maintenance has been performed, two consecutive continuing calibration check standards must pass criteria, before analysis can continue. If the analytical system can not pass two consecutive checks, then the system must be recalibrated.

G. Calibration Calculations:

1. Calculation of the relative response factor (RRF):

$$RRF=\frac{[A(x)\times C(is)]}{[A(is)\times C(x)]}$$

Where:

A(x) = Area of the characteristic ion for the compound being measured

A(is) = Area of the characteristic ion for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression equations

1st Order (linear) regression: Y = M(X) + B
2nd Order (quadratic) regression: Y = B + M(X) + CX2

Where:

Y = Conc Std / Conc Istd

X = Area Std / Area Istd

M = 1st degree slope

C = 2nd degree slope

B = Y intercept

3. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

C(c) = Measured concentration using selected quantification method

4. Calculation of the percent relative standard deviation (%RSD):

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

Where:

SD = Standard deviation

$\overline{RF}$ = Average response factor

H. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The sample component relative retention time should compare within ±0.06 RRT units of the RRT of the standard component.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The primary and secondary ions can be found in Table 4.

I. Quantitative analysis

Once a compound has been identified, quantitation will be based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. The list of primary characteristic ions is listed in Table 4.

Waters:

$$Concentration\ (\mu g\ /\ L) = \frac{A(x) \times I(s) \times V(t) \times D_f}{A(is) \times RRF \times V(o) \times V(i)}$$

Where:

A(x)	=	Area of characteristic ion for compound being measured
I(s)	=	Amount of internal standard injected (ng)
V(t)	=	Volume of concentrated extract in microliters (µL)
D_f	=	Dilution factor
A(is)	=	Area of characteristic ion for the internal standard
RRF	=	Relative response factor for the compound being measured
V(j)	=	Volume of extract injected (µL)
V(o)	=	Volume of water extracted (mL)

Soils:

$$Concentration\ (\mu g\ /\ kg) = \frac{A(x) \times I(s) \times V(t) \times G \times D_f}{A(is) \times RRF \times W(s) \times V(i) \times D}$$

Where:

A(x)	=	Area of characteristic ion for compound being measured
I(s)	=	Amount of internal standard injected (ng)
V(t)	=	Volume of concentrated extract in microliters
D_f	=	Dilution factor
A(is)	=	Area of characteristic ion for the internal standard
RRF	=	Relative Response factor for the compound being measured
V(i)	=	Volume of extract injected (µL)
W(s)	=	Weight of sample extracted or diluted in grams
D	=	The percent solids (100 - % moisture)/100
G	=	1 if extract did not require GPC cleanup
G	=	2 if extract required GPC cleanup

J. Quality Assurance:

Each extraction batch must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD) or a laboratory control sample/laboratory control sample duplicate (LCS/LCSD). Additional QC samples may be required to meet project or state certification requirements.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards	1. Peak area within -50% to +100% of the area in the associated reference standard. 2. Retention time(RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If reinjection meets the criteria, report this injection. 3. If reinjection still shows same problem, report first injection and qualify data with a comment.
Method Blank	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All target compounds must be less than the reporting limit for the associated samples.	1. Inspect system for possible problems and reanalyze. 2. If one surrogate is out of spec high and all associated sample surrogates are in spec, data can be used. (Unless project requirements dictate otherwise). * 3. If the method blank contains target analytes and the associated samples do not contain these compounds, no corrective action is required. If the target compounds in the blank are also in the associated samples, the samples should be reextracted unless it does not interfere with project data requirements.
Laboratory Control Sample/Laboratory Control Sample Duplicate	All percent recoveries within QC limits. Refer to the GC/MS Semivolatile SOP manual for QC windows. These are reviewed on a semiannual basis and updated annually.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and *reanalyze*. 3. If LCS/LCSD still out of spec, consult supervisor immediately. Samples may need to be re-extracted.

Quality Control Item	Acceptance Criteria	Corrective Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1. % Recoveries within QC limits. Refer to the GC/MS Semivolatile SOP manual for QC windows. These are reviewed on a semiannual basis and updated annually. 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries or RPDs out of spec, consult supervisor.
Surrogates	All recoveries must be within QC limits. Refer to the GC/MS Semivolatile SOP manual for surrogate windows. These are updated on a semiannual basis.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If no problem is found, reextract and reanalyze the sample.

* Requires approval of supervisor and completion of Non-Conformance Form #2586.

K. Dilution Criteria

1. Initial Dilutions:

   a. More than three internal standard areas are less than -50%.

   b. Either of the last two internal standard areas are less than -80%.

   c. Prescreen data or analyst's judgement of a sample extract's color or viscosity, indicate a possible matrix interference.

2. Secondary Dilutions:

   Are required to bring all target compounds in the calibration range of the GC/MS.

L. QC Calculations

Percent Recovery:

$$\%Recovery = Concentration\ found \div Concentration\ spiked \times 100$$

Calculations for MS/MSD:

$$Matrix\ spike\ recovery = SSR \times SR \div SA \times 100$$

Where:

SSR = Spike sample result

SR = Sample result

SA = Spike added

Relative Percent Difference (RPD)

$$RPD = \left\{ \left| MSR \times MSDR \right| \div \tfrac{1}{2} \left(MSR \times MSDR \right) \right\} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Dup Recovery

Revision Log:

Ver. #	Effective Date	Change
00	07/03/95	Previous issue
01	07/07/97	Major changes are as follows: • Added section on Personnel Training and Qualifications • Updated method references • Deleted Additional TCLP Requirements section • Removed QC Windows from tables
02	03/11/99	Major changes are as follows: • Changed method number from Analysis #0949, 0968, 1198, 1199, 1200, 1309, 1310, 1311, 1312, 1424, 1425, 1426, 4688, 4689, 4678, 4679, 4615, 4616, 4617, 4618, 3349, 7820, 7821, 7822, 7823, 7804, 7805, 5749, 5750, 7357, 7358, 7437, 7438, 7588, 7589 to Analysis #0949, 0968, 1198, 1199, 1200, 1309, 1310, 1311, 1312, 1424, 1425, 1426, 4688, 4689, 4678, 4679, 4615, 4616, 4617, 4618, 3349, 7820, 7821, 7822, 7823, 7804, 7805, 5749, 5750, 7357, 7358, 7437, 7438, 7588, 7589, 7804, 7805 • Changed title • Made changes to Reference section • Made changes to Procedure C., Standardization

Ver. #	Effective Date	Change
03	05/17/01	Major changes are as follows: • Removed analysis # 3349, 7820, 7821, 7822, 7823, 5749, 5750, 7357, 7358, 7437, 7438, 7588, 7589 • Added the use of the 25-uL syringe for internalization of standards and sample extracts • Inserted Procedure C., Instrument Conditions • Added cross-reference to Method 8000B for non-linear calibration calculations • Added Table 3 (List of compounds and associated internal standard • Added Table 4 (List of compounds with primary and secondary characteristic ions.
04	DEC 3 0 2004	Major changes are as follows: • Updated to level 3 • Added the following sections: Interferences; Sample Preservation and Holding Time; • Added Waste Handling to the Safety Precautions section. • Incorporated PA's #1 through #4 • Added ICV to initial calibration section • Added alternating concentration levels to continuing calibration section

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Prepared by: _____ Date: 12/29/04
Group Leader I

Approved by: _____ Date: 12/29/04
GC/MS Semivolatiles Management

Approved by: _____ Date: 12/30/04
Quality Assurance

Table 1

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30% to 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40% to 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5% to 9% of mass 198
275	10% to 30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17% to 23% of mass 442

Table 2

CCCs

Acenaphthene
1,4-Dichlorobenzene
Hexachlorobutadiene
Diphenylamine*
Di-n-octylphthalate
Fluoranthene
Benzo(a)pyrene
4-Chloro-3-methylphenol
2-Nitrophenol
Phenol
Pentachlorophenol
2,4,6-Trichlorophenol

Note: Diphenylamine cannot be separated from N-nitroso-di-phenylamine under the chromatographic conditions used for sample analysis.

SPCCs

N-Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

Table 3

SEMIVOLATILE INTERNAL STANDARD WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethylphenylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d5 (surr)	1-Naphthylamine
N-Nitroso-di-n-propyl amine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d6 (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
1,4-Dioxane	1-Methylnaphthalene	4-Nitrophenol
Pyridine	O,O,O-triethylphosphorothioate	Pentachlorobenzene
Acetophenone	Hexachlorpropene	1,2,4,5-Tetrachlorobenzene
o-Toluidine	1,4-Phenylenediamine	2,3,4,6-Tetrachlorophenol
N-Nitrosomethylethylamine	Safrole	2,4,6-Tribromophenol (surr)
N-Nitrosodiethylamine	(2-Bromoethyl)benzene	2,4,6-Trichlorophenol
N-Nitrosopyrrolidine	Caprolactam	2,4,5-Trichlorophenol
N-Nitrosomorpholine	1, 3, 5 – Trichlorobenzene	1,1'-Biphenyl
N,N-dimethyl formamide	1, 2, 3 – Trichlorobenzene	Diphenyl ether
N,N-dimethyl acetamide	1, 2, 3, 4 – Tetrachlorobenzene	Isosafrole
Benzaldehyde	1 – Chloro-4-Nitrobenzene	1,4-Naphthoquinone
		1,4-Dinitrobenzene
		1,3-Dinitrobenzene
		Thionazin
		5-Nitro-o-toluidine

(surr) = surrogate

Table 4

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ions
2-Picoline	93	66,92
Aniline	93	66,65
Phenol	94	65,66
Bis(2-chloroethyl) ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene-d_4 (IS)	152	150,115
1,4-Dichlorobenzene	146	148, 113
Benzyl alcohol	108	79,77
1,2-Dichlorobenzene	146	148, 113
N-Nitrosomethylethylamine	88	42,43,56
Bis(2-chloroisopropyl) ether	45	77, 121, 79
Methyl methanesulfonate	80	79,65,95
N-Nitrosodi-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
N-Nitrosodiethylamine	102	42,57,44,56
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	107	122, 121
Bis(2-chloroethoxy)methane	93	95,123
Benzoic acid	105	122,77
2,4-Dichlorophenol	162	164,98
Ethyl methanesulfonate	109	79,97,45,65
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d_8 (IS)	136	68
Naphthalene	128	129,127
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141, 115
2-Methylphenol	108	107,77,79,90
Hexachloropropene	213	211, 215, 117, 141
Hexachlorocyclopentadiene	237	235,272
N-Nitrosopyrrolidine	100	41,42,68,69
Acetophenone	105	71,51,120
4-Methylphenol	108	107,77,79,90
2,4,6-Trichlorophenol	196	198,200

Table 3 (continued)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Fluoranthene	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Hexachlorobenzene	p-Dimethylaminoazobenzene	Indeno(1,2,3-cd)pyrene
N-Nitrosodiphenylamine	Pyrene	Di-n-octylphthalate
Pentachlorophenol	Terphenyl-d_{14} (surr)	3-Methylcholanthrene
Pentachloronitrobenzene	7,12-Dimethylbenz(a)anthracene	
Phenacetin	Chlorobenzilate	
Phenanthrene	2-Acetylaminofluorene	
Pronamide	3,3'-Dimethylbenzidine	
1-Nitronaphthalene	4,4'-Methylenebis(2-Chloroaniline)	
1,2-Diphenylhydrazine		
Carbazole		
Tetraethyldithiopyrophosphate		
1,3,5-Trinitrobenzene		
Diallate trans/cis		
Phorate		
Dimethoate		
Methyl parathion		
Parathion		
4-Nitroquinoline-1-oxide		
Methapyrilene		
Isodrin		
Atrazine		

(surr) = surrogate

Table 4 (continued)

Compound	Primary Ion	Secondary Ions
o-Toluidine	106	107,77,51,79
3-Methylphenol (as 4-Methylphenol)	108	107,77,79,90
2-Chloronaphthalene	162	127,164
N-Nitrosopiperidine	114	42,55,56,41
1,4-Phenylenediamine	108	80,53,54,52
1-Chloronaphthalene	162	127,164
2-Nitroaniline	138	92, 65
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
2,6-Dinitrotoluene	165	63,89, 121
Phthalic anhydride	104	76,148
3-Nitroaniline	138	108,92
Acenaphthene-d_{10} (IS)	164	162,160
Acenaphthene	153	154, 152
2,4-Dinitrophenol	184	63, 154, 107
2,6-Dinitrophenol	162	164,126,98,63
4-Chloroaniline	127	129,65,92
Isosafrole	162	131,104,77,51
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89, 182
4-Nitrophenol	109	139,65
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	104,102,76,130
Diethyl phthalate	149	177,150
Fluorene	166	165,167
N-Nitrosodi-n-butylamine	84	57,41,116,158
4-Chlorophenyl phenyl ether	204	206,141
4,6-Dinitro-2-methylphenol	198	51, 105, 182, 77
N-Nitrosodiphenylamine	169	168,167
Safrole	162	104,77,103,135
Diphenylamine	169	168,167
1,2,4,5-Tetrachlorobenzene	216	214,179,143,218
1-Naphthylamine	143	115,89,63
4-Bromophenyl phenyl ether	248	250,141
2,4,5-Trichlorophenol	196	198,97,132,200
Hexachlorobenzene	283	142,249
Pentachlorophenol	266	264,268
5-Nitro-o-toluidine	152	77,79,106,94
Thionazin	107	96,97,143,79

Table 4 (continued)

Compound	Primary Ion	Secondary Ions
4-Nitroaniline	138	65,108,92,80
Phenanthrene-d_{10} (IS)	188	94,80
Phenanthrene	178	179,176
Anthracene	178	176,179
1,4-Dinitrobenzene	168	75,50,76,92
1,3-Dinitrobenzene	168	76,50,75,92
Diallate (cis or trans)	86	234,43,70
Pentachlorobenzene	250	252,248,215,254
5-Nitro-o-anisidine	168	79,52,138,153,77
Pentachloronitrobenzene	237	142,214,249,295
4-Nitroquinoline-1-oxide	190	160, 116, 114
Di-n-butyl phthalate	149	150,104
2,3,4,6-Tetrachlorophenol	232	131,230,166,234
Fluoranthene	202	101, 203, 100
1,3,5-Trinitrobenzene	213	74,75,120,91
Benzidine	184	92,185
Pyrene	202	101,203
Phorate	75	121,97,93,260
Phenacetin	108	179,109,137,80
Dimethoate	87	93,125,143,229
4-Aminobiphenyl	169	168,170,115
Pronamide	173	175,145,109,147
Dinoseb	211	163,147,117,240
Disulfoton	88	97,89,142,186
Butyl benzyl phthalate	149	91,206
Methyl parathion	109	125,263,79,93
Dimethylaminoazobenzene	225	120,77,148,42
Benz(a)anthracene	228	229,226
Chrysene-d_{12} (IS)	240	120,236
3,3'-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
Parathion	109	97,291, 186
Bis(2-ethylhexyl) phthalate	149	167,279
3,3'-Dimethylbenzidine	212	106,196,180
Methapyrilene	97	58, 72, 191, 261
Isodrin	193	66, 195, 263, 265,
Di-n-octyl phthalate	149	167,43, 150
2-Aminoanthraquinone	223	167, 195, 139
Aramite	185	191,319,334,197,321

Table 4 (continued)

Compound	Primary Ion	Secondary Ions
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Chlorobenzilate	139	251, 253, 111, 141
Benzo(a)pyrene	252	253,125
Perylene-d_{12} (IS)	264	260,265
7,12-Dimethylbenz(a)anthracene	256	241,239,120
2-Acetylaminofluorene	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	231	266, 140, 195
3-Methylcholanthrene	268	252,253,126,134
Indeno(1,2,3-cd)pyrene	276	138,227
Dibenz(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
1,2-Diphenylhydrazine	77	105, 182, 51
Endosulfan I	195	33
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64, 92
Nitrobenzene-d_5 (surr)	82	128,54
N-Nitrosodimethylamine	74	42,44
Phenol-d_6 (surr)	99	42,71
Terphenyl-d_{14} (surr)	244	122,212
2,4,6-Tribromophenol (surr)	330	332,141
N,N-dimethyl formamide	73	44,42
N,N-dimethyl acetamide	87	72,44,42
(2-Bromoethyl)benzene	184	77,91,105,186
Atrazine	200	173,215
Benzaldehyde	77	105, 106
Caprolacatam	113	55,56
1,1-Biphenyl	154	153,152,76
Carbazole	167	166,139
1,3,5-Trichlorobenzene	180	182,145,109
1,2,3-Trichlorobenzene	180	182,145,109
1,2,3,4-Tetrachlorobenzene	216	214,218,179
1-Chloro-4-Nitrobenzene	157	111,75,99

IS = internal standard
surr = surrogate

Analysis of Pesticides and Polychlorinated Biphenyls (PCBs) in Solid Samples

Reference:

1. Method 8081A/8082, *Test Methods for Evaluating Solid Waste*, SW-846, December 1996 (Update III).

2. Method 8081, *Test Methods for Evaluating Solid Waste*, SW-846, December 1996 (Update II).

3. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
Analysis #0173, 6030	Analysis of Polychlorinated Biphenyls (PCBs) in Aqueous Samples
Analysis #6006SON	Sonic Probe Extraction Procedure for the Determination of Pesticides in a Solid Matrix
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
SOP-OE-004	Cleanup Procedures for Pesticides Organic Extractions
SOP-PP-002	QC Data Acceptability and Corrective Action
SOP-PP-011	Interpretation of Chromatographic Data
SOP-PP-013	Preventative and Corrective GC Maintenance
SOP-PP-025	Monitoring of QC Data Acceptance Limits
SOP-PP-031	Setting Up Single Component Initial Calibrations
SOP-PP-032	Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
SOP-PP-040	Common Equations Used During Chromatographic Analyses

Scope:

This method is used for identifying and quantitating the following Pesticides and PCBs in solid samples:

Compound	LOQ (µg/kg)	Analysis
Aroclor 1016	17	1224,1225,1363,1866
Aroclor 1221	17	1224,1225,1363,1866
Aroclor 1232	17	1224,1225,1363,1866
Aroclor 1242	17	1224,1225,1363,1866
Aroclor 1248	33	1224,1225,1363,1866
Aroclor 1254	17	1224,1225,1363,1866
Aroclor 1260	33	1224,1225,1363,1866
alpha-BHC	0.83	1224,1225,1363,1866, 6000, 6001, 6005
beta-BHC	0.83	1224,1225,1363,1866, 6000, 6001, 6005
delta-BHC	0.83	1224,1225,1363,1866, 6000, 6001, 6005
heptachlor	0.83	1224,1225,1363,1866, 6000, 6001, 6005
aldrin	0.83	1224,1225,1363,1866, 6000, 6001, 6005
heptachlor epoxide	0.83	1224,1225,1363,1866, 6000, 6001, 6005
endosulfan I	0.83	1224,1225,1363,1866, 6000, 6001, 6005
endosulfan II	1.7	1224,1225,1363,1866, 6000, 6001, 6005
endosulfan sulfate	1.7	1224,1225,1363,1866, 6000, 6001, 6005
dieldrin	1.7	1224,1225,1363,1866, 6000, 6001, 6005
endrin	1.7	1224,1225,1363,1866, 6000, 6001, 6005
4,4'-DDE (*p,p*)	1.7	1224,1225,1363,1866, 6000, 6001, 6005
2,4'-DDE (*o,p*)	1.7	1363
4,4'-DDD (*p,p*)	1.7	1224,1225,1363,1866, 6000, 6001, 6005
2,4'-DDD (*o,p*)	1.7	1363
4,4'-DDT (*p,p*)	1.7	1224,1225,1363,1866, 6000, 6001, 6005
2,4'-DDT (*o,p*)	1.7	1363
endrin aldehyde	1.7	1224,1225,1866, 6000, 6001, 6005, 1363
endrin ketone	1.7	1225, 6000, 1363

Compound	LOQ (µg/kg)	Analysis
methoxychlor	8.3	1224,1225,1363, 6000, 6001, 6005
technical chlordane	17	1224,1363, 6001, 6005, 1363
alpha-chlordane	0.83	1225, 6000, 1363
gamma-chlordane	3	1225, 6000, 1363
toxaphene	33.	1224,1225,1363,6000, 6001, 6005
kepone	7	1866, 6001, 1363
mirex	1.7	1363
telodrin	0.83	1363
hexachlorobenzene(HCB)	0.83	1363

The calibrations for the PCBs following Method 8082 and for the pesticides following 8081A are run together in the same sequence when analysis for both PCBs and pesticides is requested. Since some of the PCB peaks may coelute or overlap with the pesticide peaks of interest, the joint calibration allows for better interpretation of the peaks observed for each sample. Quantitation of the same peak for two different parameters can be avoided. If a sample is clean, one run should be used to cover both methods. If there is significant matrix interference, then a separate, sulfuric acid treated fraction of extract can be run to identify and quantitate the PCBs, thereby taking advantage of the cleanups offered in 8082.

This method is based on Update III methods. See **Appendix I** for minor differences when Update II methods must be referenced.

The extraction phase of this method requires approximately 1-hour per sample with 24 samples prepared in an 8- to 9-hour shift. Each extract requires about 30 minutes to chromatograph and may require further cleanup by florisil, GPC, or copper if interferents such as oxygenated organics, unsaturated organics, or elemental sulfur are present. Refer to SOP-OE-004 for details on each cleanup procedure. Refer to extraction method 6006SON.

Analyses #6000, 6001, 6005 are for a specific client's work only to meet contractual obligations. The numbers are identical to existing pesticide scans except they are for pesticides only—no PCBs are included (6000 = 1225; 6001 = 1866; 6005 = 1224).

Only the TCL list of organochlorine pesticides will be reported for OH VAP work.

Basic Principles:

A 30-g portion of sample is extracted using sonication with 50% hexane/acetone. The extract is dried, concentrated, and exchanged to hexane. The pesticides/PCBs are then identified and quantitated using gas chromatography with electron capture detector. Florisil, GPC, or copper cleanups may be employed to reduce matrix interferences which introduce large, unresolvable peaks into the chromatogram, specifically elemental sulfur.

Personnel Training and Qualifications:

Each analyst performing the instrumental analysis will work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform calculations, interpret data, and enter data into the LIMS. They will also follow the department training manual for analysts. Proficiency is measured through documented audits of the tasks listed and overchecking of data as well as annual quad studies.

Interferences:

An electron capture detector is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur. Plastic should not be used during the extraction or analysis to prevent phthalate contamination. Glassware must be scrupulously cleaned. Florisil cleanup is used to reduce other organics which can interfere (polar compounds). GPC is used to remove sulfur and higher molecular weight

organics. Copper cleanup is used to remove elemental sulfur. All of these materials can introduce large, unresolvable peaks into the chromatogram.

A 1:1 mixture of methylene chloride/acetone has been shown to introduce many extraneous interfering peaks due to reactions that occur between the solvents during the heated concentration process. Therefore, hexane/acetone is used.

Safety Precautions and Waste Handling:

See extraction 6006 for those related to sample prep and handling.

Gloves, lab coats, and safety glasses should be worn when preparing standards. Lab coats and safety glasses should be worn around the GC where solvents and sample extracts are handled.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Apparatus and Equipment:

1. HP 5890 gas chromatograph equipped with dual electron capture detectors or equivalent

2. Columns:

   RTX - CLPesticides I - 30 m × 0.32 mm × 0.5 µm
   RTX - CLPesticides II - 30 m × 0.32 mm × 0.25 µm

3. Integrating system such as Chrom Perfect by Justice Innovations or equivalent

4. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards:

A. Reagents

1. Hexane for autosampler rinse vials

2. UPC (ultra pure carrier) helium for carrier gas

3. UPC nitrogen for detector make-up gas

4. UPC hydrogen for carrier, either bottled or from a generator

B. Standards:

All standards are prepared using Class A volumetric pipettes, flasks, and syringes.

1. PCB standards are identical to those outlined in Analysis #0173, 6030.

2. Mix A – Restek Catalog #32292. Equivalent to the CLP (SOW OLMO3.2) Mix A and B, contains all single component pesticides and surrogates in the TCL and PPL organochlorine lists.

3. Mix E – Restek Custom Mix #55992. Contains additional organochlorine pesticides such as kepone, the *o,p* isomers of DDT, DDD, DDE, mirex, and telodrin.

4. Toxaphene stock – Restek Catalog #32005 at 1,000,000 ppb. Prepare an intermediate by placing 1 mL into a 10-mL volumetric and bring to volume with hexane.

5. Technical chlordane stock – Ultra Scientific PP-150, 100,000 ppb in methanol.

6. Surrogate Stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetra-chloro-meta-xylene (TCX) at 200,000 ppb each in acetone.

7. Pest MS stock – Supelco Catalog #48796, #48196. All compounds in Mix A (#2 above) at various concentrations.

8. EVAL stock – Restek 32074-510. Equivalent to CLP performance evaluation mix.

9. ICV stocks – These should always be from different lot numbers (or vendors) than the working calibration standards. Mix A and B, Restek Catalog #32297 and #32298. Prepare a five-fold dilution by adding 1 mL of each stock to a 5-mL volumetric and bring to volume.

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description	Expiration Date
SW-846 SS	SS Stock	1.5	1000	acetone or methanol	SW-846 Water Surrogate - identical to that prepared for PCB analyses	6 months
SW-846 MS	MS Stock	1.0	50	acetone or methanol	SW-846 Water Spike for single component organochlorines in the TCL/PPL list	6 months
Mix A Intermediate	Restek 32292	1.25	25	hexane		6 months
MIXA1	Mix A Intermediate	0.125	100	hexane	Mix A Level 1 calibration	6 months
MIXA2	Mix A Intermediate	0.5	100	hexane	Mix A Level 2 calibration	6 months
MIXA3	Mix A Intermediate	2	200	hexane	Mix A Level 3 calibration	6 months
MIXA4	Mix A Intermediate	2.5	100	hexane	Mix A Level 4 calibration	6 months
MIXA5	Mix A Intermediate	5	100	hexane	Mix A Level 5 calibration	6 months
Mix E Intermediate	MIX E Stock	.25	25	hexane		6 months
SS Intermediate	TCX/DCB Stock	0.25				

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description	Expiration Date
MIXE1	Mix E Intermediate	0.2	100	hexane	Mix E level 1 calibration	6 months
	SS Intermediate	0.2				
MIXE2	Mix E Intermediate	0.5	100	hexane	Mix E level 2 calibration	6 months
	SS Intermediate	0.5				
MIXE3	Mix E Intermediate	1.6	200	hexane	Mix E level 3 calibration	6 months
	SS Intermediate	2.5				
MIXE4	Mix E Intermediate	2	100	hexane	Mix E level 4 calibration	6 months
	SS Intermediate	2				
MIXE5	Mix E Intermediate	4	100	hexane	Mix E level 5 calibration	6 months
	SS Intermediate	4				
TOXAX	Toxaphene Intermediate	0.25	50	hexane	Toxaphene calibration - single level	6 months
	SS Intermediate	0.5				
TOXA1	Toxaphene Intermediate	0.5	50	hexane	Toxaphene calibration – level 1	6 months
	SS Intermediate	0.4				
TOXA2	Toxaphene Intermediate	0.5	25	hexane	Toxaphene calibration – level 2	6 months
	SS Intermediate	0.4				
TOXA3	Toxaphene Intermediate	2.5	50	hexane	Toxaphene calibration – level 3	6 months
	SS Intermediate	0.8				
TOXA4	Toxaphene Intermediate	2.5	25	hexane	Toxaphene calibration – level 4	6 months
	SS Intermediate	0.8				
TOXA5	Toxaphene Intermediate	5	25	hexane	Toxaphene calibration – level 5	6 months
	SS Intermediate	1.6				

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description	Expiration Date
CHLD1	Chlordane Stock	0.025	100	hexane	Technical Chlordane calibration level 1	6 months
	SS intermediate	0.2				
CHLD2	Chlordane Stock	0.05	100	hexane	Technical Chlordane calibration level 2	6 months
	SS intermediate	0.1				
CHLD3	Chlordane Stock	0.2	200	hexane	Technical Chlordane calibration level 3	6 months
	SS intermediate	4				
CHLD4	Chlordane Stock	0.2	100	hexane	Technical Chlordane calibration level 4	6 months
	SS intermediate	3				
CHLD5	Chlordane Stock	0.5	100	hexane	Technical Chlordane calibration level 5	6 months
	SS intermediate	4				
EVALX	EVAL Stock	1	100	hexane	Breakdown check mix - identical to the CLP Performance Evaluation Mix(PEM)	6 months
	SS Intermediate	1				
MDLA	Mix A Level 1	10	50	hexane	Mix A MDL Standard	6 months
MDLE	Mix E Level 1	10	50	hexane	Mix E MDL Standard	6 months
MDTXX	TOXAX	2	50	hexane	Toxaphene MDL Standard	6 months
MDCHX	CHLDX	1.25	50	hexane	Chlordane MDL Standard	6 months
MD16X	AR16 Level 1	10	50	hexane	1016/1260 MDL Standard	6 months
MD21X	AR21 Level 1	10	50	hexane	1221 MDL Standard	6 months
MD32X	AR32 Level 1	10	50	hexane	1232 MDL Standard	6 months
MD42X	AR42 Level 1	10	50	hexane	1242 MDL Standard	6 months
MD48X	AR48 Level 1	10	50	hexane	1248 MDL Standard	6 months

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description	Expiration Date
MD54X	AR54 Level 1	10	50	hexane	1254 MDL Standard	6 months
ICV Intermediate	Second Source Stocks	1	5	hexane	Intermediate	6 months
ICMAX	ICV Intermediate	2.5	250	hexane	ICV Second Source Check Standard	6 months
	SS Intermediate	5				

NOTE: All solutions are stored in the freezer at 15° ± 5°C. Unopened ampules are store at room temperature.

Waste Management:

All GC vials and vials containing extracts are placed in a hazardous waste container for lab pack disposal. There is a satellite container in the laboratory that is then emptied into the main laboratory waste collection drums. All solvent waste is disposed of in solvent waste containers.

Extraction:

See Organic Extraction Method 6006.

Sample Preservation and Holding Time:

Samples are collected in 125-mL wide-mouth glass containers with Teflon-lined caps and kept cool at 2° to 4°C. Samples are homogenized prior to the extraction. The extraction must be performed within 14 days of collection, and sample analysis must be performed with 40 days of extraction.

Gas Chromatographic Conditions:

Detector – ECD

Detector temp – 300°C

Oven temp – 140°C, no hold time, 15°C/min to 300°C, hold for 6 min

Carrier – Hydrogen at 8.7 PSI (helium may also be substituted)

Makeup gas – N_2 at 30 mL/min for Varian GCs, 55 mL/min for HP GCs

Injection size – 1 µL, direct injection

Injector temp – 220°C

The conditions listed are usually the optimum operating conditions but may vary to improve the sensitivity, linearity, and overall chromatography or shorten run times on each GC system. A Merlin microseal may be used in place of a traditional septum.

EPA Method Deviation: GC conditions differ from those listed in 8081. However, all QC criteria are met.

Gas Chromatographic Analysis:

1. Prior to starting a new calibration, change the septum on the GC unless no more than 200 injections have been made or a Merlin is being used and allow the system to stabilize. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.

2. Prepare a sequence using the following order of injections:

    1. Conditioner
    2. EVALX
    3. MIXA1
    4. MIXA2
    5. MIXA3
    6. MIXA4

7. MIXA5
8. MIXE1*
9. MIXE2*
10. MIXE3*
11. MIXE4*
12. MIXE5*
13. TOXAX
14. CHLDX
15. AR161
16. AR162
17. AR163
18. AR164
19. AR165
20. AR21x
21. AR32x
22. AR42x
23. AR483
24. AR543
25. MDLAX
26. MDLEX
27. MDTXX
28. MDCHX
29. MD16X
30. ICMAX
31. ICV for 1016/1260
32. Blank
33. LCS
34. 1234567
35. 1234567MS
36. 1234567MSD

37. - 47. Continue running samples for a 12-hour period from last standard

48. EVALX
49. MIXA3
50. MIXE3*
51. AR163

*MIXE is only needed when analyzing for Analysis #1866 (for kepone) and #1363.

Continue running groups of samples/injections followed by check standards every 12 hours or 20 injections, whichever comes first. The EVALX standard is run to check analyte breakdown. Single component pesticide check standards are rotated among MIXA level 2, 3, and 4, followed by a rotation of AR162, 3, or 4 for an aroclor check. Instrument blanks (IBLK) may also be run with the continuing check standards—this is optional but frequently requested for projects.

For projects where a known aroclor is present and at the request of clients, other aroclors can be run for the continuing check standard.

The conditioner injection is usually a standard or sample that has already been injected. It is used to prime the system and is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections. Hexane blanks may also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

3. The GC is calibrated using the five levels of the single component pesticides contained in MIXA and E, aroclors 1016, 1260 and using the single point for chlordane, toxaphene, 1221, 1232, 1242, 1248, and 1254. An external standard calibration is used with average calibration factor (AVGCF) for all analytes where the %RSD is ≤20%. If the average of the %RSDs of all compounds in the initial calibration standard is ≤20%, the AVGCF may be used for all compounds in the initial calibration. Alternatively, when these criteria are not met, a calibration curve will be used. A linear fit will be tried first. However, if the correlation coefficient is <0.99, a quadratic fit will be tried. A 6-point calibration must be run to use quadratic. Prepare a sixth point somewhere within the established calibration range listed in the standards preparation section.

If toxaphene or technical chlordane is detected in a sample, the sample will be rerun along with a full five-point calibration for that analyte and check standards.

The curve will not be forced or extrapolated to zero, rather the mathematical intercept will be used for quantitation. (It should be noted that this can result in false positives or negatives for peak heights below the low-point standard, depending on where the intercept falls.) See SOP-PP-031 for more details on using the data system generating forms and general calibration information.

If the 0.99 curve coefficient cannot be met, inspect the data points to see if one or more calibration levels appear to be off. A specific calibration level may have concentrated due to solvent evaporation or degraded over time. Reinject or remake the standard if this is the cause. Otherwise, the instrument may need maintenance. See SOP-PP-013 for troubleshooting linearity problems.

Curve types and criteria can be altered to meet client or project specific requirements, as well as any regulatory agency requirements that may differ from those listed here.

A quadratic fit may not be used for South Carolina samples. Additionally, use of the average of the %RSDs (grand mean) for using the AVGRF as the calibration fit is not permitted for samples from South Carolina.

4. The aroclor calibration data is setup in a custom program called datalog. The retention time of the peaks to use for identifying and quantifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations. See SOP-PP-032 for details on using datalog. The calibration is an external standard using the AVGCF. The PCBs must meet the 20% RSD criteria.

5. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections which contain them.

6. The scaling and peak integration of chromatograms parameters should be set so that the peaks of interest for each compound are detected and integrated at the concentration of the method detection limit (MDL). This ensures that the quantitation limits and MDLs can be met.

7. Calibration verification includes a breakdown check, alternating levels of single-component calibration standards and alternate multi-component standards. After each set of injections in a 12-hour period, or 20 samples, whichever comes first, (samples, QC, blanks, etc.), a set of check standards must be run. Alternating concentration levels of MIXA are run to evaluate the range of the calibration for the single component analytes, and of AR16 for the aroclors. The low and high points from the calibration standards are not included in the rotation due to the potential for the response for these to fall outside the calibration range. The concentration quantitated for the continuing calibration check standards must be within 15% difference (%D) of the nominal concentration. Samples must be bracketed by compliant standards. If confirmation of target analytes is needed, then the second column must meet the 15% continuing calibration criteria, as well as all initial calibration criteria.

   Any samples which follow a non-compliant standard must be rerun after a standard that meets the 15% D criteria, or after a new initial calibration.

   If the average of the %Ds meets ±15%, the standard is considered compliant. If an individual compound is outside the 15% criteria but the standard passes using the average of the %Ds, any sample results with hits for that compound must contain a comment stating this situation occurred. The experience of the analyst should be used to determine if the sample should be reinjected after a check standard that is not within 15% for the target compound.

Use of the average of the %Ds (grand mean) for continuing calibration standards is not permitted for samples from South Carolina.

8. Retention time (RT) windows are established as 3× the standard deviation determined over 72 hours, or at no less than 0.03 minutes, applied to the initial calibration standard, usually Level 5. If the RTs for a continuing calibration standard fall outside the RT window, update the midpoint RT using that standard. Save this under the appropriate name to indicate an update has occurred. All subsequent standards run within a 24-hour period must be within this window. Retention times cannot be updated more than once per day. If RTs are not consistent, the cause should be investigated and corrective action taken.

9. Retention times of peaks in the samples are compared to the standard RT windows. Peaks present on both columns (and that are also in the correct ratios to represent an aroclor) are quantitated, and the high value is reported unless there are chromatographic anomalies. See SOP-PP-011.

10. If significant interference is present, schedule florisil cleanup. If elemental sulfur is present, copper cleanup the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract and adjust LOQs accordingly.

Calculations:

See SOP-PP-040 for details on all calculations/equations used to evaluate the initial and continuing calibration. Calculation of results is performed according to the following procedures:

A. Single-component compounds

1. Using AVGCF from initial calibration:

$$\frac{\textit{Sample Height}}{\textit{AVG CF}} \times \frac{FV}{IW} \times DF = \textit{µg/kg as received}$$

2. Using linear curve from initial calibration:

$$[(\textit{Sample Height} - Y\text{-}\textit{intercept}) / \textit{Slope}] \times \frac{FV}{IW} \times DF = \textit{µg/kg as received}$$

Where:

FV (final volume) = 10 mL

IW (initial weight) = 30 g

DF (dilution factor) = as needed

B. Multi-component compounds

The peak heights generated by the integration system are used to calculate the response factors (RF) for peaks of interest for each aroclor. Usually the six major peaks that are unique to each aroclor are chosen for quantitation with the exception of 1221 where only three peaks are available. Sample concentrations are calculated per peak using Average Calibration Factor (AVG CF).

$$\frac{\textit{Sample Height}}{\textit{AVG CF (CF)}} \times \frac{FV}{IW} \times DF = \textit{µg/kg as received}$$

Where:

FV (final volume) = 10 mL

IW (initial weight) = 30 g

AF (additional factor) = based on cleanups
(i.e., 2 mL extract florisiled to 25 mL = 12.5)

DF (dilution factor) = as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(Result\ 1 + Result\ 2 + \ldots + Result\ n) / n = \text{Average } Result$$

NOTE: If toxaphene or technical chlordane is detected in a sample, the sample will be rerun along with a full five-point calibration for that analyte and a check standard. If an aroclor other than 1016/1260 is detected, quantitation is performed against the single point standard that was part of the initial calibration. The response for the individual peaks in the sample aroclor pattern must be within the response of the upper level calibration standard of 1016/1260. If they exceed, then a dilution must be performed. Method 8082 allows this approach. The 1016/1260 standards are used to assess the linearity of the aroclor peaks. Method 8082 does not require a five point calibration for any aroclor detected. A five point calibration can be performed for other aroclors as needed to meet project specific requirements or other agency or regulatory requirements.

C. A breakdown mix (EVALX) containing p,p'-DDT and Endrin is run to check for breakdown. The breakdown should not exceed 15% for either compound. Breakdown is calculated as:

$$\%\ Breakdown\ for\ p,p'\text{-}DDT = \frac{pk\ ht\ (area)\ of\ p,p'\text{-}DDE + p,p'\text{-}DDD}{pk\ ht\ (area)\ of\ p,p'\text{-}DDE + p,p'\text{-}DDD + p,p'\text{-}DDT} \times 100$$

$$\%\ Breakdown\ for\ Endrin = \frac{pk\ ht\ (area)\ of\ Endrin\ Aldehyde + Endrin\ Ketone}{pk\ ht\ (area)\ of\ Endrin\ Aldehyde + Endrin\ Ketone + Endrin} \times 100$$

If breakdown fails, injector maintenance must be performed. Analysis cannot proceed until breakdown check passes.

Statistical Information/Method Performance:

Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are generated according to LOM-SOP-ES-203. MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. These are run on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Results from all instruments are compared and pooled together to determine the reporting MDL. Copies of the annual studies are maintained by the department supervisor. Updates to the LIMS are made as needed by the QA department and only as directed by the supervisor. The department database is updated via a download from the LIMS. Each analyst has hard copies of the current reporting limits for each analysis.

Quality Assurance/Quality Control:

A deionized water blank and deionized water spike (LCS) are analyzed with every group of samples up to a maximum of 20. An MS/MSD is performed per batch as long as there is ample volume of a sample in the batch. If an MS/MSD cannot be performed, an LCSD will be extracted. All single-component pesticides of interest for each analysis

are routinely spiked with the exception of analysis 1363. Mirex, *o,p*-DDE/DDD/DDT, telodrin, kepone, and HCB are not spiked since this would result in co-elution with the other spiked compounds. These can be spiked at a client's request for special projects.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

See SOP-PP-002 for details on QC acceptance criteria and corrective action.

QC Acceptance limits are generated according to SOP-PP-025.

Revision Log:

Ver. #	Effective Date	Change
00	07/17/96	Previous Issue
01	01/15/98	Major changes are as follows: • Entire method rewritten for organochlorine pesticides/PCBs only • Removed prep • Analysis number changed from #1216, 1224, 1225, 1363, 0819, 1866, 1867, 5367, 4854, 6000, 6001, 6005, 6624, 6677, 6678 to #1363, 1224, 1225, 1866, 4854, 6624, 6000, 6001, 6005
02	06/16/99	Major changes are as follows: • Add Update II method reference • Add Appendix I Update II requirements • Add hydrogen and Merlin microseal options • Change batching requirements

Ver. #	Effective Date	Change
03	05/18/01	Major changes are as follows: • Added Cross Reference section • Added references to SOP-PP-040 • Clarified QC section • Changed RF to CF • Added storage conditions for standards
04	06/13/03	Major changes are as follows: • Incorporated PA #1 - breakdown calculation • Interference section added • ICV standard added • CCV criteria updated • Added copper cleanup • Added info on 5 pt curve for multi's in calculation section • Updated to Level 3 format • *Changed prep # to 6006 and reference hexane/acetone* • Delete 6624 • Updated standards, GC conditions • Add Waste Management section
05	04/22/04	Major changes are as follows: • Updated method
06	MAY 1 1 2006	Major changes are as follows: • Updated scope – LOQs • Added requirement and stds scheme for full 5-pt curve for toxaphene and chlordane • Incorporated PA #1 in Gas Chromatographic Analysis section – added additional information to items 3 and 7

COMPANY CONFIDENTIAL
Level 3 Document

Prepared by: _____ Date: 4/24/6
Senior Chemist

Approved by: _____ Date: 4/25/06
Pesticide Residue Analysis Management

Approved by: Barbara Bleedy Date: 4/27/06
Quality Assurance

Appendix I
SW 846 Update Requirements

If a client or agency requires the use of Update II method references for a given sample(s), the following changes to the method as written need to be made:

ICAL:
If any individual component's %RSD is >20%, a calibration curve rather than the average RF must be used. The curve fit must meet a .99 correlation coefficient to be valid. A quadratic fit does not require the sixth point. The average of the %RSDs for all compounds cannot be used to justify using an average RF for compounds exceeding 20%RSD.

CCAL:
The %D for each individual component must meet 15%D to be compliant. The average of the %Ds cannot be used to determine if a continuing calibration standard is compliant. If samples are not bracketed by compliant check standards, they must be reinjected.

Second Column:
The second column can be used for qualitative confirmation if the CCAL does not meet the 15% criteria on one of the columns. If there is a hot on the column that meets 15% and the second column shows increasing sensitivity and confirms or negates the presence of the compound, the data can be reported. However, if the second column shows decreasing sensitivity and does not confirm the presence of the compound, the sample should be reinjected since there is a significant chance for a false negative. All hits should be reported from the column meeting the 15%D criteria.

Aroclor Hits:
If an aroclor is detected in a sample that has only been calibrated using a single concentration level, the sample must be rerun with a 5-point calibration for that particular aroclor.

Uncontrolled Copy

Synthetic Precipitation Leaching Procedure
SPLP Nonvolatile Leachates

Reference:

1. Method 1312, *Test Methods for Evaluating Solid Waste*, SW-846, USEPA, September 1994.

2. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
LOM-SOP-ES-225	Reagents and Standards
LOM-SOP-LAB-220	Laboratory Notebooks, Logbooks, and Documentation
SOP-TL-001	Glassware Cleaning for Leachate Extractions
SOP-TL-002	Leachate Blank Evaluations
SOP-TL-003	Subsampling and Preservation of Leachates

Scope:

This method is used to determine the mobility of organic and inorganic nonvolatile contaminants in potentially hazardous waste. The extraction is performed over 18 hours.

Personnel Training and Qualifications:

The technician using this method should be trained by a qualified technician and perform these procedures at least twice in that person's presence before they may be considered qualified.

Interferences:

Any potential interferences that may be encountered during the analysis are discussed in the individual analytical methods.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Follow routine safety steps. Special care must be taken when mixing the acid solution. Wear gloves, lab coats, and safety glasses when preparing the solution and handling samples. Read all lab notes and follow instructions carefully. Avoid skin contact or breathing the vapors from any of the reagents or samples. Use appropriate ventilation system (bench top or hood) when instructed, if the sample looks suspicious (plating waste contains cyanide), or is particularly odoriferous.

If the sample fumes or reacts in any other way when acid is added during the extraction procedure, vent the sample periodically until the reaction is no longer evident.

Discard or send for repair any glassware that is chipped, flawed, or broken.

Sample Handling:

Samples should not be preserved and should be stored at 2° to 4°C. The hold time for performing the leachate extraction is 14 days from the time of collection to the day the extraction is begun.

Evaluate the solid waste for particle size. The material should be capable of passing through a 9.5-mm sieve or have a diameter of <1 cm upon visual inspection. If the particle size does not meet this criteria, have the sample pulverized (if it is stone-like material) or cut the material into smaller pieces if possible.

Glassware Cleaning:

See SOP-TL-001.

Method Summary:

For liquid wastes (containing <0.5% solids), the SPLP extract is defined as the filtrate resulting from filtration of the waste through a 0.6- to 0.8-µm glass fiber filter.

For wastes containing >0.5% solids (and some liquid), the waste is filtered through a glass fiber filter and the filtrate is stored for later use. The solid is then extracted with a volume of extraction fluid 20× the weight of the solid. Following the extraction, the leachate is filtered through a glass fiber filter and the final and initial filtrates are combined.

For waste which will obviously yield no liquid when subjected to pressure filtration, the sample is extracted with a volume of appropriate extraction fluid at 20× the weight of the sample. The extract is then filtered and the leachate is defined as the filtrate.

Apparatus and Equipment:

1. Agitation apparatus (tumbler) – This must be an EPA-approved device that is capable of rotating the extraction vessels at 30 ± 2 rpm in an end-over-end manner

2. Extraction vessel – This should be a jar large enough to contain the volume of extraction fluid required (within the limitation of tumbler space); the vessel should be made of borosilicate glass, Teflon™, or polyethylene

3. Filtration device – The Millipore Hazardous Waste Filtration system is suitable (also known as pressure filters)

4. Vacuum filtration system – Consisting of a vacuum flask, filter holder, and hose

5. Acid washed Glass fiber filters (0.6- to 0.8-µm pore size) – Assorted sizes

6. pH meter – Orion Model 210A, or equivalent; capable of 0.01 pH unit display

7. Graduated cylinders – Class A, assorted sizes

8. Glass jar for combining extracts

9. Glass bottles – Assorted sizes

10. Jug for preparing extraction fluid

11. Pipettes – Class A, assorted sizes

12. Volumetric flasks – Class A, assorted sizes

13. Gooch crucible

14. Balance – Capable of weighing to 0.01 g

Reagents and Standards:

All reagents must be labeled and documented in accordance with LOM-SOP-LAB-220 and LOM-SOP-ES-225.

1. SPLP stock solution (60%H_2SO_4/40%HNO_3)

Sulfuric acid, concentrated (ACS reagent grade), H_2SO_4	6 mL

Nitric acid, concentrated (ACS reagent grade), HNO_3	4 mL

Measure 6 mL of H_2SO_4 into a graduated cylinder and pour into a 100-mL volumetric, containing approximately 50 mL of deionized water. Measure 4 mL HNO_3 into a graduated cylinder and pour into the same volumetric. Bring to volume with deionized water. Store at room temperature. Remake every 6 months.

2. SPLP Intermediate

SPLP Stock	10 mL

Using a graduated cylinder, add 10 mL of SPLP stock solution to a 1000-mL volumetric flask containing approximately 500 mL of deionized water. Dilute the solution to volume with deionized water and mix thoroughly. Store at room temperature in a glass bottle for up to 6 months.

3. Extraction Fluid #1 (East and West non-soils)

SPLP Intermediate	20 mL

Using a graduated cylinder, add 20 mL of SPLP intermediate to 10 L of deionized water. Mix thoroughly. The pH of this fluid must be 4.20 ± 0.05. Record the preparation and the pH of the fluid in the appropriate logbook. Make enough for use each day and discard any extra.

NOTE: This extraction fluid is to be used with samples that come from east of the Mississippi River. In addition, this fluid is to be used for samples west of the Mississippi that are not soils.

4. Extraction Fluid #2 (West Soils)

SPLP Intermediate	3 mL

Using a graduated cylinder, add 3 mL of SPLP intermediate to 10 L of deionized water. Mix thoroughly. The pH of this fluid must be 5.00 ± 0.05. Record the preparation and the pH of the fluid in the appropriate logbook. Make enough for use each day and discard any extra.

NOTE: This extraction fluid is to be used with **soil** samples that come from west of the Mississippi River.

5. Extraction Fluid #3

This fluid is deionized water and is used for waste or soils that are being extracted for cyanide. This must be used for cyanide-containing samples because under acidic conditions, hydrogen cyanide gas may result.

6. 1N nitric acid

Nitric acid, concentrated (ACS reagent grade), HNO_3	64 mL

Using a graduated cylinder, slowly add 64 mL of concentrated HNO_3 to approximately 500 mL deionized water in a 1000-mL volumetric flask. Dilute the solution to volume with deionized water. Remake every 6 months. Store in a glass bottle at room temperature.

7. Nitric acid, 5N

Nitric acid, concentrated ACS reagent grade, HNO_3	320 mL

Using a graduated cylinder, slowly add 320 mL of concentrated nitric acid to approximately 500 mL deionized water in a 1000-mL volumetric flask. Dilute the solution to volume with deionized water. Remake every 6 months. Store in a glass bottle at room temperature.

Determining the Amount of Extract to Prepare:

1. Look up the sample in the Parallax computer system under the sample data program.

2. Note the analyses requested. Information about the sample requirements can be found in Table I.

3. Add up the sample volume required and divide by 20 to determine how much sample must be extracted.

4. If the amount of extraction fluid generated by a single SPLP extraction will not be sufficient to perform all analyses, more than one extraction may be performed and the leachate from each extraction must be combined and then aliquoted for analysis.

Preliminary Solids Determination:

If the waste appears to be liquid and seems to contain a very low percentage of solids, perform the solids determination. The Federal Register TCLP method defines percent solids as that fraction of a waste sample from which no liquid may be forced out by an applied pressure.

1. Preweigh the filter in a gooch crucible and record the weight in the percent solids determination section of the nonvolatile prefilter spreadsheet (see Figure 1).

2. Weigh out a subsample of the waste.

a. Weigh out 100 ± 0.1 g of sample into a graduated cylinder if there is adequate sample for the leachate and other analyses required. If not, use proportionally less in the graduated cylinder. Record the sample number and the weight of the sample plus the graduated cylinder on the spreadsheet.

b. Pour the sample into the vacuum filter apparatus and slowly apply vacuum until no liquid flows through the filter.

c. Reweigh the graduated cylinder – Record the weight of the graduated cylinder and residue adhered to the cylinder. The spreadsheet will calculate the weight of sample filtered.

3. The material in the filter holder is defined as the solid phase (sludge cake and filter) of the waste and the liquid phase is the filtrate.

4. Weigh the sludge cake and filter and record the weight on the spreadsheet.

5. The Spreadsheet will determine the percent solids:

$$\%Solids = \frac{Weight\ of\ solid\ recovered}{Weight\ of\ sample\ filtered} \times 100$$

6. If the percent solids is <0.5%, print the spreadsheet and go to Procedure A.

7. If the percent solids is between 0.5% and 2.0%, dry the filter and solid at 100° to 120°C until two successive weighings yield the same value within ±1%. Record each weight on the spreadsheet. The percent dry solids are determined using the following calculation:

$$\%DrySolids = \frac{Weight\ of\ dried\ solid\ recovered}{Weight\ of\ sample\ filtered} \times 100$$

8. If the percent dry solids is <0.5%, print the spreadsheet and go to Procedure A. If the percent dry solids is ≥0.5%, go to Procedure B.

Procedure:

A. If the sample contains <0.5% solids:

1. Filter the waste through a glass fiber filter (0.6- to 0.8-µm). The filtrate is defined as the SPLP extract.

2. Using a pH meter, determine and record the pH of this extract in the logbook.

3. Change the analysis from #1567 to #1339 in Parallax. Enter the appropriate comments and preserve the subsamples in accordance with SOP-TL-003 and Table I.

B. If the sample contains ≥0.5% solids and has a standing liquid phase:

1. Weigh the filter paper that will be used for the prefilter. Record the weight of the filter in the prefilter section of the nonvolatile prefilter spreadsheet (see Figure 1).

2. Weigh a minimum of 100 g of sample in a graduated cylinder. Record the weight of the sample and cylinder on the spreadsheet.

3. Pour the sample into the pressure filter. Weigh the empty cylinder and record the weight on the spreadsheet.

4. Slowly apply vacuum to the pressure filter assembly beginning with 10 psi. As the rate of filtration decreases, increase the pressure in 10 psi increments. Continue to increase the pressure until the maximum pressure of 50 psi is reached. Stop the filtration when no filtrate appears over a 2-minute interval or when air is being forced through the filter.

5. If the layer of liquid in the solid is organic and does not mix with water, a large quantity of sample will need to be filtered. This is because each layer of the final leachate (Procedure B, step 15) will have to be analyzed separately and enough solids will need to be collected to generate enough extraction fluid for the assigned analyses.

6. When filtration is complete, transfer the solids and filter into a tared beaker and weigh. Record the weight on the spreadsheet.

7. Using a graduated cylinder, measure the volume of the initial filtrate and record this figure on the spreadsheet.

8. Transfer the solids and filter from step 6. above to an extraction vessel. It may be necessary to reduce the particle size of the solid to <1 mm in diameter before placing in the vessel.

9. Using a graduated cylinder, slowly add the appropriate extraction fluid at 20× the weight of the solid.

   NOTE: If the sample contains cyanide, use Extraction Fluid #3. If the sample is a **soil** from a site west of the Mississippi River, use Extraction Fluid #2. If the sample is from a site east of the Mississippi River, or a **non-soil** from west of the Mississippi river, use Extraction Fluid #1.

10. Close the extraction bottle tightly and place in a tumbler (rotating 30 ± 2 rpm) for 18 ± 2 hours. Record the ID of the tumbler and start time in the leachate extraction logbook. Ambient temperature must be maintained at 23°± 2°C (69.8° to 77°F) during the extraction period.

11. Following the 18 ± 2 hour extraction, filter the sample through a glass fiber filter, again taking care to increase the pressure in 10-psi increments and not to exceed 50 psi if using the pressure filter. This filter can be changed if necessary.

12. Record the volume of extract fluid recovered from the sample.

13. The spread sheet will calculate the amount of initial filtrate to add back, using the following calculation:

$$\textit{Volume of initial filtrate to be added} = \frac{\textit{Volume of extraction fluid collected (mL)}}{\textit{Volume of extraction fluid (mL) added}} \times \frac{\textit{Weight of sample extracted (g)}}{\textit{weight of sample recovered (g)}} \times \textit{mL of initial filtrate}$$

14. Print the spreadsheet.

15. If the initial filtrate does not mix (is immiscible) with the extraction fluid, do not mix the two together. Contact a supervisor and technical services. Each phase must be analyzed separately; therefore, each phase will be entered as a sample.

16. Using a pH meter, determine and record the pH of the leachate in the logbook.

17. Using pipettes, add the appropriate matrix spike to metals samples. In order to give the correct spike levels, 4 mL of each spike (A and B) should be added to 200 mL of leachate.

18. Subsample and preserve the leachate for the appropriate analyses in accordance with SOP-TL-003 and Table I.

C. If the sample has no freestanding liquid:

1. Reduce a portion of the solid to a particle size of <1 cm in diameter, if necessary.

2. Weigh out a minimum of 100 ± .1 g of the waste (more if a larger volume of leachate is required for analysis).

3. Using a graduated cylinder, slowly add the appropriate extraction fluid at 20× the weight of the solid.

   NOTE: If the sample contains cyanide, use Extraction Fluid #3. If the sample is a **soil** from a site west of the Mississippi River, use Extraction Fluid #2. If the sample is from a site east of the Mississippi River, or a **non-soil** from west of the Mississippi river, use Extraction Fluid #1.

4. Close the extraction bottle tightly and place in a tumbler (rotating 30 ± 2 rpm) for 18 ± 2 hours. Record the ID of the tumbler and start time in the leachate extraction logbook. Ambient temperature must be maintained at 23° ± 2°C (69.8° to 77°F) during the extraction period.

5. Following the 18 ± 2 hour extraction, filter the sample through a pressure filter assembled with two stacked glass fiber filters of the same pore size (one 125mm and one 150mm), again taking care to increase the pressure in 10-psi increments and not to exceed 50 psi if using the pressure filter. This filter can be changed if necessary. If multiple jars of the sample were leached, combine the extracts.

6. Using a pH meter, measure and record the pH of the extract in the logbook.

7. Using pipettes, add the appropriate matrix spike to metals samples. In order to give the correct spike levels, 4 mL of each spike (A and B) should be added to 200 mL of leachate.

8. Subsample and preserve the leachate for the appropriate analyses in accordance with SOP-TL-003 and Table I.

Quality Assurance/Quality Control:

1. A new matrix batch must be prepared each day samples are leached. Batches may not be continued. Therefore, each matrix is a new batch each day.

2. An extraction fluid tumble blank must be prepared for each extraction fluid used per day. A blank must be started for each 20 samples with the same fluid type.

3. A blank will be performed after every 20th time an individual extraction vessel is used. A logbook of vessel usage is kept. Each time a vessel is used, record the date and the sample number. After 20 uses, prepare a blank in that vessel and record the blank number in the logbook. Every blank will be evaluated for contamination using the guidelines in SOP-TL-002.

4. Record all the sample numbers in the batch logbook. One sample in the batch should be designated as the waste-type spike and spiked in accordance with the notes in Table I. Each subsample type in the batch must have a spike. Record the lot number of the spike solution used on the batch sheet. Photocopy the sheet and deliver the photocopies to each department involved in the analyses. Enter the appropriate leachate information in Parallax.

5. Extraction of the solid phase should be initiated as soon as possible after initial filtration.

6. All instruments used in this procedure (i.e., pH meter and balance) should be calibrated according to an approved laboratory plan.

7. All quality control measures described in the appropriate analytical methods shall be followed.

Revision Log:

Ver. #	Effective Date	Change
00	08/06/96	Previous Issue
01	08/06/98	Major changes are as follows: • Update procedure • Added Personnel Training and Qualifications section
02	03/05/99	Major changes are as follows: • Incorporated Procedural Amendment #1 • Updated metals preservation and spiking portion of procedure • Added items 1 and 2 to Quality Assurance section
03	08/27/01	Major changes are as follows: • Cross Reference – Section added • Apparatus – Clarified • Reagents – Clarified preparation • Glassware Cleaning – Section added • Preliminary Solids Determination/Procedure – Added information about entering data in nonvolatile prefilter spreadsheet, clarified pressure increases • Quality Assurance – Clarified • Table I – Updated • Figure 1 – Added
04	12/31/01	Major changes are as follows: • Reagent section – Added preparation of SPLP Intermediate solution and changed preparation of extraction fluids
05	07/17/02	Major changes are as follows: • Updated use of Extraction fluids 1 and 2 • Clarified pressure increases during filtration

Ver. #	Effective Date	Change
06	07/22/04	Major changes are as follows: • Updated document to Level 3 format • Clarified use of acid washed filters • Removed references to Wang computer system
07	MAY 16 2005	Major changes are as follows: • Removed references to pre-filters in Apparatus and Equipment section • Clarified sizes of acid washed filters • Minor wording changes throughout

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Prepared by: Darin Wagner Date: 4/19/05
Senior Chemist, Group Leader

Approved by: [signature] Date: 4-22-05
Organic Extraction Management

Approved by: Dorothy McKee Date: 5/2/05
Quality Assurance

Table I

Leachate Scheduling/Preservation			
Prep Analysis/Name	817/Pest	813/3337/7807 Semi	5705 Metals
Bottle Code	31	43	8
Preferred Vol	2000	2000	500
Minimum Vol	1000	1000	100
Preservation	None	None	HNO_3
Departments	36/24	36/26	23

NOTES: CRG metals – BKG 500 mL, spike 200 mL for sample QC
Semi spike (matrix) – 4 × 1000 mL
Pest spike – 4 × 1000 mL
Metals spike – 200 mL

Figure 1

Sample # 1234567
Date:
Technician:

Percent Solids Determination

Liquid/Solid Separation	
Weight of filter paper and gooch (g)	1.00
Weight of sample and grad	1.00
Weight of grad and residue	1.00
Weight of sample filtered	0.00
Weight of solid plus filter/gooch (g)	1.00
Weight of solid recovered (g)	0.00
Percent Solids	#DIV/0!
Weight of dried sample and filter	1.00
Weight of dried sample and filter	1.00
Weight of dried sample and filter	1.00
Weight of dried sample	0.00
Percent of dried solids	#DIV/0!

Non-Volatile Leachate Prefilter

Liquid/Solid Separation	
Weight of filter paper (g)	1.00
Weight of sample and grad	1.00
Weight of grad and residue	1.00
Weight of sample (total)	0.00
Weight of solid plus filter (g)	1.00
Weight of solid recovered (g)	0.00
Percent Solids	#DIV/0!
Weight of sample extracted (g)	0.00
Volume of ext. fluid to add (mL)	0
Volume of initial filtrate (mL)	1
After Tumble	
Volume of ext. fluid collected (mL)	1
Volume of initial filtrate to add (mL)	#DIV/0!

Uncontrolled Copy

Analysis of Chlorinated Herbicides in Soil

Reference:

1. Method 8151A, *Test Methods for Evaluating Solid Waste*, SW-846, Update III, December 1996.

2. Method 8151, *Test Methods for Evaluating Solid Waste*, SW-846, Update II.

3. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
Analysis #0816	Extraction of Chlorinated Herbicides in a Water Matrix
Analysis #4181	Extraction of Chlorinated Herbicides in Soil Matrix
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
SOP-OE-004	Cleanup Procedures for Pesticides Organic Extractions
SOP-PP-002	QC Data Acceptability and Corrective Action
SOP-PP-011	Interpretation of Chromatographic Data
SOP-PP-025	Monitoring of QC Data Acceptance Limits
SOP-PP-031	Setting Up Single Component Initial Calibrations
SOP-PP-040	Common Equations Used During Chromatographic Analyses

Scope:

This method is used for identifying and quantitating the following chlorinated herbicides in soils and solids.

Compound	Limit of Quantitation (µg/kg)
Dalapon	60
Dicamba	5
MCPP (Mecoprop)	2500

Compound	Limit of Quantitation (µg/kg)
MCPA	2500
2,4 - DP (Dichloroprop)	17
2,4 - D	17
2,4,5 - TP (Silvex)	1.7
2,4,5 - T	1.7
2,4 - DB	17
Dinoseb	8.3
Pentachlorophenol	1.7
Hexachlorophene	10

This method is based on Update III methods. See Appendix I for minor differences if Update II methods must be referenced. The extraction phase of this method requires approximately 12 hours for one technician to prepare eight samples. The extract requires 40 minutes to chromatograph if hexachlorophene is one of the analytes of interest. If hexachlorophene does not need to be chromatographed, the extract can be run in 20 minutes. A dilution may be required if interferences such as chlorinated acids and phenols are present. A florisil cleanup is included to remove some interferences. Refer to SOP-OE-004 for more details on this cleanup procedure. Refer to extraction method 0816.

This method is used for analyzing soil and sediment samples scheduled for Lancaster Laboratories Analysis #1863, 1865, 0344, 5592 and Analysis #4181. See Table I for a list of Compounds in each analysis number.

Basic Principles:

The compounds of interest are extracted with 1:1 methylene chloride:acetone from an acidified portion of soil (pH less than 2) using sonication. The extract is hydrolyzed and interfering compounds like chlorinated hydrocarbons and phthalates are removed by a solvent wash. After acidifying the extract once more, the compounds are extracted with ethyl ether and are converted to their methyl esters using diazomethane as the derivatizing agent. The methyl esters are determined using gas chromatography with an electron capture detector. A florisil cleanup is performed to eliminate matrix interferences that introduce large, unresolvable peaks in the chromatogram.

Personnel Training and Qualifications:

Each analyst performing the instrumental analysis will work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret the chromatograms, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and overchecking of data, as well as annual quad studies.

Interferences:

An electron capture detector is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur. Plastic should not be used during the extraction or analysis to prevent phthalate contamination. Glassware must be scrupulously cleaned. All of these interferents can introduce large, unresolvable peaks into the chromatogram. Florisil cleanup is used to reduce other organics which can interfere (polar compounds). Additionally, the extraction incorporates a solvent wash step to remove potential interfering organic compounds.

Safety Precautions:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

See Analysis #4181 for those related to sample preparation and handling.

Gloves, lab coats, and safety glasses should be worn when preparing standards. Safety glasses should be worn around the GC where solvents and extracts are handled.

Apparatus and Equipment:

1. HP5890 gas chromatograph fitted with electron capture detector, or equivalent

2. RTX – CLPesticides – 30 m × .0.32 mm × 0.5 µm

3. RTX – CLPesticides II – 30 m × .0.32 mm × 0.25 µm

4. Integrating system such as Chrom Perfect from Justice Innovations or equivalent

5. Various sizes of Class A volumetric pipettes, flasks, and syringes.

Reagents and Standards:

All standards are prepared using Class A volumetric pipettes, flasks, and syringes.

A. Reagents

1. Hexane, pesticide grade for autosampler vials

2. UPC (ultra pure carrier) nitrogen for detector make up

3. UPC helium for carrier gas

4. UPC hydrogen, bottled or from a generator

B. Standards

1. Herb stock – Methylated herbicide stock for calibration standards: Ultra HBM8150M (concentrations vary per compound)

2. DCAA stock – 2,4-dichlorophenylacetic acid (DCAA) methylated stock: Ultra PPS-161 (100,000 ppb)

3. PCP inter – Make a 100-fold dilution of pentachlorophenol stock Ultra PH-180 (100,000 ppb) into methanol. Methylate this solution to prepare a 10,000 ppb methylated intermediate.

4. DBOB stock – 4,4-dibromooctafluorobiphohenyl Ultra PPS-170 (100,000 ppb) used as internal standard

5. DCAA SS stock – 2,4-dichlorophenylacetic acid. Non-methylated stock. Ultra PSS-162 (5,000,000 ppb)

6. MS stock – Herbicide mix in the acid form for the matrix spiking solution: Ultra HBM8150A (concentrations vary per compound).

7. Hexachlorophene stock – Prepare a 1,000,000-ppb stock from neat material, Chem Service F995, by diluting 0.025 g into 25 mL of methanol. This solution remains in the acid form. Alternatively, a solution can be purchased if it is available at this concentration.

8. Hexachlorophene intermediate – Dilute 1 mL of hexachlorophene stock into 100 mL of hexane. This solution is methylated following the procedure outlined in Analysis #0816 or Analysis #4181.

9. ICV stock mix – Accustandard M8150/51 in isooctane. Varied concentrations.

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description	Expiration Date
Herb 1	Herb Stock	0.05	100	Hexane	Level 1 Calibration	6 months
	DCAA Stock	0.05				
	PCP Inter	0.05				
	DBOB Stock	0.1				
Herb 2	Herb Stock	0.1	100	Hexane	Level 2 Calibration	6 months
	DCAA Stock	0.1				
	PCP Inter	0.1				
	DBOB Stock	0.1				
Herb 3	Herb Stock	0.4	200	Hexane	Level 3 Calibration	6 months
	DCAA Stock	0.4				
	PCP Inter	0.4				
	DBOB Stock	0.2				
Herb 4	Herb Stock	0.4	100	Hexane	Level 4 Calibration	6 months
	DCAA Stock	0.4				
	PCP inter	0.4				
	DBOB Stock	0.1				
Herb 5	Herb Stock	1	100	Hexane	Level 5 Calibration	6 months
	DCAA Stock	1				
	PCP Inter	1				
	DBOB Stock	0.1				
SS	DCAA SS Stock	0.4	200	Acetone or Methanol	Herb Water Surrogate	6 months
MS	MS Stock	2.5	100	Acetone or Methanol	Herb Water Matrix Spike	6 months
	PCP Stock	0.05				
MS-DBOB	DBOB Stock	1	10	Hexane	Internal Standard	6 months
HEXA 1	Hexachlorophene Intermediate	0.1	50	Hexane	Hexachlorophene Level 1 Calibration	6 months
	DBOB Stock	.05				
HEXA 2	Hexachlorophene Intermediate	0.25	50	Hexane	Hexachlorophene Level 2 Calibration	6 months
	DBOB Stock	.05				

Standard Name	Parent Solution	Aliquot (mL)	Final Vol (mL)	Solvent	Description	Expiration Date
HEXA 3	Hexachlorophene Intermediate	1.0	100	Hexane	Hexachlorophene Level 3 Calibration	6 months
	DBOB Stock	0.1				
HEXA 4	Hexachlorophene Intermediate	1.0	50	Hexane	Hexachlorophene Level 4 Calibration	6 months
	DBOB Stock	.05				
HEXA 5	Hexachlorophene Intermediate	2.0	50	Hexane	Hexachlorophene Level 5 Calibration	6 months
	DBOB Stock	.05				
MS	Hexachlorophene Stock	0.05	25	Methanol or Acetone	Hexachlorophene Water MS	6 months
MDLH	Herb 1	10	50	Hexane	MDL Standard	6 months
ICV Intermed. (methylated)	ICV stock	1.0	10	Hexane	ICV Second Source intermediate – methylate following procedure in method 816	6 months
	DCAA stock	0.02				
ICHBX	ICV intermed	0.2	10	Hexane	ICV Second Source Working Standard	6 months
	DBOFB Stock	0.01				

Sample Preservation and Holding Times:

The holding time for this extraction is 14 days from collection. The extracts must be analyzed within 40 days of extraction. Samples must be stored at 2° to 4°C. There is no preservation. However, samples should be homogenized prior to extraction.

Waste Disposal:

All GC vials are disposed of in the designated waste container in the lab, then subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the lab, then emptied into the lab-wide waste facility.

Procedure:

Extraction: See Analysis 4181

GC Analysis:

Instrument setup (Primary and Confirmation):

Detector – ECD
Detector Temperature – 300°C
Makeup Gas – N2 at 30 mL/min for Varian ECDs, 55 mL/min for HP ECDs
Injection Size – 2 µL, direct injection
Injector Temperature – 250°C
Oven Temperature – 85°C, hold 2 min, 30°C/min to 170°C, 10°C/min to 270°C, hold 6 mins
Carrier – Hydrogen at 10 psi (Helium may be substituted.)

The conditions listed above are optimum but may be changed to improve the linearity, sensitivity, and chromatography on each GC system. A Merlin microseal may be used in place of a traditional septum.

1. Prior to starting a new calibration, change the septum on the GC unless a Merlin is being used and allow the detector to stabilize. Fill the autosampler rinse vials with clean solvent or replace vials themselves if they appear to be dirty.

2. Prepare a sequence using the following order of injections:

    1. Conditioner
    2. Herb Level 1

3. Herb Level 2
4. Herb Level 3
5. Herb Level 4
6. Herb Level 5
7. MDLH
8. ICV
9. Blank
10. LCS
11. 1234567
12. 1234567MS
13. 1234567MSD
14. – 18. Continue with samples
19. Herb Level 3
20. – 29. 10 samples
30. Herb Level 4

Continue running groups of 10 samples with alternating herbicide levels 2, 3, and 4 as the standard between sample groups. Additionally, five levels of hexachlorophene should be run along with Herb Level 5 if analyzing for that compound.

The conditioner injection is usually a standard or sample that has already been injected. It is used to prime the system and is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections. Hexane blanks may also be run to allow the GC to go through some temperature program runs and/or to check the cleanliness of the system.

3. The system is calibrated using the five concentration levels – Herb Level 1 through 5. An internal standard calibration is used with average calibration factor (AVGCF) for all analytes where the %RSD is ≤20%. If the average of the %RSDs of all compounds in the initial calibration standard is ≤20%, the AVGCF may be used for all compounds in the initial calibration. Alternatively, when these criteria are not met, a calibration curve will be used. A linear fit will be tried first. However, if the correlation coefficient is <0.99, a quadratic fit will be tried. A 6-point calibration must be run to use quadratic. Prepare a

sixth point somewhere within the established calibration range listed in the standards preparation section. For either curve type, extrapolate or force zero is not allowed. See SOP-PP-031 for details on using Chrom Perfect for setting up single component calibration files.

4. Ensure all peaks in the standard are labeled properly and the scaling of the plot is such that concentrations at the MDL exhibit a peak about 2 to 3 mm in height. Be sure all peaks on the MDLH standard are integrated by the data system.

5. Alternating concentration levels of herbicide calibration standards (HERB level 2, 3, or 4) are run to evaluate the range of the calibration for the target analytes. The low and high points from the calibration standards are not included in the rotation due to the potential for the response for these to fall outside the calibration range. The continuing calibration check standards between samples must exhibit a response at ±15% Difference (%D) for each compound, or the average of the %Ds must be within ±15% for the standard to be compliant on at least one of the two columns used for analysis. The concentration calculated for the continuing injection is compared to the nominal concentration.

   Samples must be bracketed with compliant standards. If, however, the standard following a sample is outside the ±15% but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected. If confirmation of target analytes is needed, then the second column must meet the 15% continuing calibration criteria, as well as all initial calibration criteria.

   If a compound is outside the 15% criteria but the standard passes using the average of the %Ds, any sample results with hits for that compound must contain a comment stating this situation occurred. The experience of the analyst should be used to determine if the sample should be reinjected after a check standard that is within 15% for the target compound.

6. Retention time (RT) windows are established as 3× the standard deviation determined over a 72-hour period, or at no less than ±0.03 min, applied to the mid-point initial calibration standard. If the RTs for a continuing calibration standard fall outside the windows, update the midpoint RT using that standard. Save this under an appropriate name to indicate an update has occurred. All subsequent continuing standards run within a 24-hour period must fall within this window. RTs cannot be updated more than once per day. If RTs are not consistent, the cause should be investigated and corrective action taken.

7. Retention times of peaks in the samples are compared to the standard RT windows. Peaks that are present on both columns are quantitated and the high value is reported unless there are chromatograph anomalies. See SOP-PP-011.

8. If significant interference is present, make a dilution of the sample.

   When preparing dilutions, add sufficient internal standard to maintain the same 100-µg/L final concentration.

Calculations:

1. Linear curve

$$Sample\ Conc.\ \mu g / kg = Extract\ Conc. \times \frac{DF \times FV}{IW} \times Conc.\ Internal\ Standard$$

FV = Final volume = 100 mL

W = Initial weight = 10 g

DF = Dilution factor

Where:

$$Extract\ Conc.\ (\mu g / L) = \frac{\frac{Peak\ Height}{Internal\ Standard\ Height} - Y - intercept}{slope}$$

2. Average response factor (AVG CF)

The calculation performed by AVG CF is the same as above except the extract concentration is calculated as follows:

$$Extract\ Conc.\ (\mu g / L) = \frac{Peak\ Height\ in\ Sample}{Internal\ Peak\ Height\ in\ Sample} / AVGCF$$

Where:

$$AVG\ CF = (CF\ Calib\ 1 + CF\ Calib\ 2 + \ldots + CF\ Calib\ 5) / 5$$

$$CF = \frac{Standard\ Peak\ Height}{Internal\ Peak\ Height} / \frac{Standard\ Concentration\ (mg / L)}{Conc.\ Internal\ Standard}$$

Also see SOP-PP-040 for more details on calculations regarding the calibration.

Quality Assurance/Quality Control:

A sodium sulfate blank and laboratory control spike (LCS) are analyzed with each group of samples. Matrix spike and matrix spike duplicate are analyzed with each batch of 20 samples as long as there is ample volume. An LCSD will be performed if an MS/MSD cannot be done. The spiking solutions contain all analytes of interest. A surrogate standard of 2,4-dichlorophenyl acetic acid (DCAA) is added to each sample, blank, and spike to monitor the efficiency of the extraction and the operation of the autoinjector.

Statistical Information/Method Performance:

The QC acceptance limits for LCS, MS/MSD and surrogates are established according to SOP-PP-025. SOP-PP-002 outlines the QC acceptability criteria and corrective action.

Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are generated according to LOM-SOP-ES-203. MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. These are run on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Results from all instruments are compared and pooled together to determine a reporting MDL. Copies of the annual studies are maintained by the department supervisor. Updates to the LIMS are made as needed by the QA department and only as directed by the supervisor. The department database is updated via a download from the LIMS. Each analyst has hard copies of the current reporting limits for each analysis.

Revision Log:

Ver. #	Effective Date	Change
00	08/25/95	Previous Issue

Ver. #	Effective Date	Change
01	04/18/97	Major changes are as follows: • Added hexachlorophene • Deleted prep sections • Included information from SOP-PP-008, "Setting Up and Checking an Analytical Sequence for Samples Analyzed for Herbicides by Method 8151, SW-846" • Added standards prep section
02	01/15/98	Major changes are as follows: • LOQs • Added MDL standard • Added LCSD • Changed initial and continuing calibration to meet 8000B
03	07/12/99	Major changes are as follows: • Add Update II method and Appendix I of requirements • Add batching requirements • Add hydrogen and Merlin microseal options
04	10/03/01	Major changes are as follows: • Added Cross Reference section • Deleted columns DB608/001 • Added Apparatus • Added quad study requirement • Changed RPD to %D • Changed RF to CF • Added SOP-PP-040 t0 Cross Reference section

Ver. #	Effective Date	Change
05	11/17/03	Major changes are as follows: • Updated format to Level 3 • Updated LOQs • Rotation of CCVs • Added Interference section • Added Waste Disposal section • Added ICV criteria
06	NOV 2 2 2005	Major changes are as follows: • Added Interferences section • Added Statistical Information/Method Performance section • Updated standards prep • Updated reporting limits

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Prepared by: _____ Date: 10/26/05
Senior Chemist

Approved by: _____ Date: 10/26/05
Pesticide Residue Analysis Management

Approved by: _____ Date: 11/8/05
Quality Assurance

Table I

Compound	1863 (Appendix IX)	1865 (RCRA)	344	5592
2,4-D	X	X		
2,4,5-TP (Silvex)	X	X		
2,4,5-T	X	X		
Dinoseb	X	X		
Dalapon		X		
MCPP		X		
MCPA		X		
2,4-DP		X		
2,4-DB		X		
PCP		X	X	
Hexachlorophene				X

Appendix I

SW-846 Update II Requirements

If a client or agency requires the use of Update II method references for a given sample(s), the following changes to the method as written need to be made.

ICAL:

If any individual component's %RSD is >20%, a calibration curve rather than the average RF must be used. The curve fit must meet a .99 correlation coefficient to be valid. A quadratic fit does not require the sixth point. The average of the %RSDs for all compounds cannot be used to justify using an average RF for compounds exceeding 20%RSD.

CCAL:

The %D for each individual component must meet 15%D to be compliant. The average of the %Ds cannot be used to determine if a continuing calibration standard is compliant. If samples are not bracketed by compliant check standards, they must be reinjected.

Second Column:

The second column can be used for qualitative confirmation if the CCAL does not meet the 15%D criteria on one of the columns. If there is a hit on the column that meets 15%, and the second column shows increasing sensitivity and confirms or negates the presence of the compound, the data can be reported. However, if the second column shows decreasing sensitivity and does not confirm the presence of the compound, the sample should be reinjected since there is a significant change for a false negative. All hits should be reported from the column meeting the 15%D criteria.

Determination of Volatile Target Compounds by Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS) in Soils and Solids by Method 8260B

Reference:

1. Method 8260A, Revision 1, SW-846, U.S. EPA, September 1994.
2. Method 8260B, Revision 2, SW-846, U.S. EPA, December 1996.
3. Method 8000B, USEPA SW-846, Rev. 2, December 1996.
4. Method 5035, USEPA SW-846, Rev. 0, December 1996.
5. *GC/MS Volatiles Training Manual*, Current Version.
6. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
Analysis #0374	Solid Matrix Sample Preparation
Analysis #8389, 8390	Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035
MC-EX-001	GC/MS Preventative and Corrective Maintenance
SOP-MS-001	Glassware Cleaning
SOP-MS-004	GC/MS Routine and Nonroutine Maintenance
SOP-MS-006	GC/MS Volatile Standards Traceability
SOP-MS-012	GC/MS Volatiles Audit Process

Scope:

This method is suitable for the determination of compounds listed in Tables I-A through I-L for soil and solid matrices. Samples consist of soils/solids collected with methanol added (as the preservation/extraction fluid) or 'as received'. Non-target volatile compounds in the sample can be tentatively identified (TIC) using a mass spectral reference library comparison. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by purge and trap GC/MS methodologies and skilled in mass spectral interpretation. Using this method, the TICs are quantitated with an estimated concentration.

Compounds other than those listed in Tables I-A through I-L may be analyzed using USEPA SW-846 Method 8260B. Selected Ion Monitoring (SIM) parameters can be used to detect, identify and quantitate volatile organic compounds in methanol extracts of soils/solids, if lower quantitation limits are required (project- or client-specified) and/or matrix interferences are anticipated. Due to poor purging efficiency or poor chromatography, some analytes require calibration at higher levels and/or higher practical quantitation limits (PQLs). Any additional compounds should be added to the theoretical standard concentrations (TSC) sheet. Standards containing additional analytes should be prepared as described in the Standards section of this document. Both secondary stock solutions and matrix spike solutions should be prepared for use in analyzing additional compounds.

Basic Principles:

The soil sample is prepared according to Analysis #0374 for 8260A (and 8260B) and #8389, 8390 for 8260B. The sample is purged with an inert gas and the effluent gas passed through a sorbent trap where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and backflushed onto the head of a gas chromatographic column held at the appropriate initial temperature for the column in use. The gas chromatographic column is temperature programmed to separate the volatile compounds, which are subsequently detected and identified using mass spectrometric techniques.

When a compound reaches the MS, it is bombarded by high energy electrons (70 eV). This causes the compound to fragment and form ions. The positive ions are focussed into a quadrupole mass analyzer, where the ions are separated according to their mass/charge ratios during rapid repetitive scans. These ions are then amplified and detected with an electron multiplier.

The resulting time/intensity/mass spectra data are stored and processed by a computer. Target compounds are identified on the basis of relative retention times and mass spectral matches to standards, which are injected every 12 hours on the same system. The internal standard method is used for quantitation.

Personnel Training and Qualifications:

Education Requirement: A 4–year Baccalaureate Degree from an accredited College or University in one of the physical sciences and/or 1 – 3 years of relevant gas chromatography experience.

Analysts must be trained in the proper method of volatile organic sample preparation and analysis as determined by the supervisor(s). All training and education relating to volatile organic sample preparation and analysis shall be documented by each analyst in their training record. All analysts performing this method must have read this SOP.

Specifically, each new chemist will train with an experienced chemist for the first 12 weeks depending on the individual and their previous experience. The first 12 weeks are spent working one-on-one with the trainer. This time may be less if the new chemist has prior relevant experience in GC/MS and analytical chemistry background. Each new chemist receives a training manual outlining the basics of operating the instrumentation and producing usable data.

During the training period, the new chemist will learn daily maintenance, column and source changing procedures, calibration techniques, data and library search review, and forms generation. They are also required to read all relevant SOPs and EPA methods.

To measure the proficiency of each chemist, several checks have been established. The first is the ability to successfully calibrate. The chemist will run a series of at least five calibration standards and perform the calibration routine. A departmental data validator will then review the resultant curve. They will confirm that relative retention times (RRT) and response factors (RF) match throughout the calibration and ID list. Secondly, each analyst must perform a quad study. This will consist of analyzing four back-to-back replicates at a known concentration. This process will measure accuracy as well as reproducibility of results. It is a requirement that quad studies are performed by each analyst on an annual basis.

Interferences:

Contaminant sources are volatile compounds in the laboratory environment, impurities in the inert purging gas, carryover from samples containing high concentrations of volatile organic compounds and dirty glassware. The analyst must demonstrate that the system is free from interferences (by producing acceptable method blank data) before analyzing a batch of samples. Matrix effects from heavily contaminated soils and solids can interfere with the internal standard responses, target analytes and surrogate recoveries, thereby hindering accurate quantitation. See Section 3.0 of SW-846 Method 8260B for further discussion.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity of each reagent used has not been precisely determined. However, each reagent must be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves when working directly with reagents. Refer to the *Lancaster Laboratories Chemical Hygiene Plan* for specific details.

Sample Handling:

Apparatus and Equipment:

1. Gastight micro syringes – 10 µL and larger

2. 5-mL gastight syringes

3. Analytical balance, capable of accurately weighing ±0.0001 g

4. Top-loading balance capable of weighing ±0.01 g

5. Glassware

   a. Class A Volumetric flasks with ground-glass stopper

   b. Vials, 1.5 mL, 15 mL, and 40 mL screw cap, with Teflon/silicone septa

   c. Mininert vials, 1 mL, 2 mL, and 5 mL

6. *Purge and trap device* - Consisting of the sample purger, the trap, and desorber; the OI Analytical 4560, OI Analytical 4660, or equivalent meets the requirements of this method. The purging chamber should have the purge gas passing through the sample as finely divided bubbles and minimize the headspace between the sample and the trap to <15 mL.

7. Autosampler - OI Analytical 4551, Archon, or equivalent meets the requirements of this method. The Archon has the capability to purge solid samples directly in the 40-mL vial (needed for samples prepared by Analysis #8389).

8. Spiker units (optional) – OI Analytical Model 4551 SIM/Spiker or equivalent. One or two automated syringe spikers can be added to the OI Analytical Model 4551 autosampler to automatically introduce 10 µL of internal standard

(ISTD), surrogate standard, and/or matrix spiking solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of appropriate standards.

9. GC/MS system - The HP 5890GC/5970 MSD, HP 5890GC/5971 MSD, HP 5890GC/5972 MSD, HP 6890GC/5973MSD, and Shimadzu GC/MS QP5000 meet the requirements for this method.

10. Data System/Computer/Software– this is interfaced to the GC/MS system, which continuously acquires and stores data during the analysis, and can process/reduce data to generate the appropriate forms and supporting data. The software used for acquisition is HP Chemstation®, and data reduction is accomplished using Target® software.

11. GC Columns

    a. Column 1 – 75M × 0.53 mm ID DB-624 capillary column with a 3.0-µm film thickness from J&W Scientific, or equivalent

    b. Column 2 – 30M × 0.25 mm ID DB-624 capillary column with a 1.4-µm film thickness from J&W Scientific, or equivalent

Different sampling/analysis combinations are used, based on how the sample was collected, expected level of VOCs in the sample, possible matrix interferences, list of target compounds, whether TICs were requested, the required quantitation limits and the type of equipment/instrumentation available. Refer to Procedures 0374, 0890, 1178, 3982, 2657, 2658, 4276, 7157 or client specific SOPs for guidance in choosing a particular methodology.

Reagents and Standards:

A. Reagents

1. Reagent water is defined as water in which an interferant is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory will meet these criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Purge and Trap grade Methanol

B. Standards

See SOP-MS-006 for standards traceability.

1. Stock standard solutions - Stock solutions must be prepared in methanol. Standards are prepared from ampulated and neat compounds obtained from suppliers that indicate the purity of the compound. No correction for purity is made if the purity is listed as ≥96%. Pre-made solutions can be used if the supplier documents the concentrations of the solutions. All ampulated standards are stored at -10° to -20°C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided.

   NOTE: For most of the target compounds, the stock standard solutions are purchased from a vendor as custom mixes (V for calibration and Q for separate source quality control). The internal and surrogate standards are purchased from a vendor, including the target compounds that are gases at room temperature. These gaseous standards have a 1-week expiration date, starting from the date they are opened. In some applications the stock standard is used as is.

2. Surrogate stock standard solution (for high level soils) - a 2500 µg/mL stock standard solution of dibromofluoromethane, toluene-d8, 4-bromofluorobenzene, and 1,2-dichloroethane-d4 is prepared in methanol by a commercial supplier

3. Internal standards solution - a 2500 µg/mL stock standard solution of fluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4 is prepared in methanol by a commercial supplier. If an Archon autosampler is used, dilute the stock to 250 µg/mL in methanol. This is assuming a 1-µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained (50 µg/kg or 1 µg/kg for SIM scan). Deuterated tertiary butyl alcohol (tBA-d10) is used sometimes as an auxiliary ISTD. It is prepared from neat, and used as specified, depending on the instrument used and the analysis required

   To prepare stock standards from neat compounds:

   a. Place about 9.8 mL methanol or an equivalent solvent into a tared 10.0-mL glass-stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

   b. Add the liquids using a syringe or pipette by adding 2 or more drops of the assayed material to the flask, being careful that no drop hits the side of the flask. Bring the volume of solvent in the flask to 10.0 mL. Calculate the concentration of the standard.

   c. The stock standard solutions are stored in Teflon-sealed screw-capped vials at -10° to -20°C. The compound name, concentration, date prepared, and expiration date must appear on the bottle.

   d. Replace in house prepared stock standard solutions every 6 months.

4. Secondary dilution standards - Using the stock standard solutions, prepare secondary stock solutions in methanol containing the desired compounds. Tables I-A through I-L list the compounds analyzed under Analysis #2304, 2308.... These standards are prepared by calculating the volume of each stock standard required producing a given volume of a mixed working standard with a known concentration of each analyte. When custom mixes

are used, these may be diluted down individually, or combined together with other mixes. The working standard is tested according to SOP-MS-006. The verified working standard is poured into Teflon-lined screw-capped GC vials or mininert vials and stored at -10° to -20°C. A designator indicating the standard, month, and day of preparation must be on the standard vials. The designator and the calculations for the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every 6 months.

a. 1,4-Bromofluorobenzene (BFB) standard - Prepare a 25-µg/mL solution of BFB in methanol by diluting the stock standard (prepared from neat material) with methanol to a final volume of 100 mL. The volume of stock standard used will vary depending on the actual stock concentration.

b. Surrogate standard spiking solution - Prepare the surrogate standard spiking solution from the stock standard solutions at a concentration of 25 or 250 µg/mL in methanol.

c. Internal standard (IS) solution (for ICAL use) - fluorobenzene, t-butyl alcohol d_{10} (if needed), chlorobenzene-d5, and 1,4-dichlorobenzene-d4. One mL of 8260IS is diluted with methanol to a total volume of 10 mL to give a final concentration of 250 µg/mL. This is assuming a 1 µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained.

d. IS/SS spiking solution – Dilute 1 mL of 8260IS and 1 mL of 8260SS with methanol to 10 mL final volume (resulting in a concentration of 250 µg/mL). This is assuming a 1 µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained. If tBA-d10 is to be used, the resulting concentration should be 1250 µg/mL.

e. Calibration spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. Suggested calibration levels are 4, 10, 20, 50, 100, and 300

f. Matrix spiking solution - Prepare solutions in methanol that contain the compounds of interest at known concentrations. These solutions serve as both the matrix spiking solution and the laboratory control sample solutions. Matrix spikes also serve as duplicates; therefore, two aliquots of the same sample need to be spiked for analysis with these solutions. Replace the matrix spiking solution every 6 months.

g. Store all standard solutions at -10° to -20°C.

Procedure:

A. Daily maintenance: Refer to SOP-MS-004

B. Instrument Conditions:

1. The purge and trap device should have the trap conditioned for at least 10 minutes at 180° to 220°C at a flow rate of 20 to 60 mL/min prior to initial use.

2. An example of purge and trap conditions are listed below:

Purge gas	Helium
Purge flow	35 - 40 mL/min
Purge temperature	40°C for low level soils and ambient temp. for medium/high level soils
Purge time	11 min
Desorb temperature	190°-220°C
Desorb time	0.5 to 4 minutes **
Bake temperature	180°-220°C
Bake time	8 min (±3 min)

** - Range as suggested by the purge and trap instrument manufacturer

NOTE: Purge and trap conditions may be changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument may be found in the appropriate instrument maintenance log.

3. The suggested gas chromatographic operating conditions are listed in the table below, depending on the column used:

	Column 1	Column 2
Column liquid phase	DB-624	DB-624
Carrier gas	Helium	Helium
Carrier gas flow	9-10 mL/min	.8 mL/min
Make-up gas flow	25 mL/min	None
Initial temperature	35°C	45°C
Initial hold time	5 min	4.5 min
Temperature ramp	10°C/min	12°/min to 100°C then 25°/min to 240°C
Final temperature	180°C	240°C
Final hold time	5 min	None

4. Recommended mass spectrometer (MS) operating conditions:

Ions	Positive
Electron energy	70 volts
Mass range	35 - 300 amu

H-P systems Scan time:

Number A/D Samples	2^2 (4)
Integration Time/Sample	50 microsec
Total Scan Time	0.6 sec

5. Recommended MS operating conditions for SIM scan of target compounds :

SIM Parameters

Group	Ions	Time Range	Target Compounds
1	57, 73, 78, 102, 111, 113	4.00-6.30	Dibromofluoromethane, MTBE
2	52, 70, 77, 78, 96, 102, 113	6.30-8.50	1,2-Dichloroethane-d4, Benzene, Fluorobenzene,
3	91, 92, 95, 106, 117, 174	8.50-14.00	Toluene-d8, Toluene, Chlorobenzene-d5, Ethylbenzene, m+p-Xylene, o-Xylene, 4-Bromofluorobenzene

NOTE: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions that give the performance required by the method are acceptable.

C. Tuning:

Tune the GC/MS system to meet the criteria in Table II following a 50-ng injection of BFB. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the temperature ramp may be increased and the initial temperature and flow rate may be different. The BFB tune must be verified every 12 hours.

The tune must be evaluated by taking the average of the three scans across the BFB peak apex with a background subtraction of a scan within 20 scans prior to the start of the BFB peak.

NOTE: All standards, samples, and associated quality control samples must be analyzed with the same mass spectrometer parameters as those used to obtain a successful tune.

D. Initial Calibration:

1. Perform the initial calibration by analyzing at least six distinct levels of analyte and surrogate concentrations and producing response factors for each compound (six levels are required if second order regression fits are used). (Refer to SOP-EX-001 for the preparation of calibration standards). The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of the analyte concentration.

2. A method detection limit (MDL) standard must be analyzed with each initial calibration. This standard is prepared at or near the departmental MDL and is not to be included in the calibration curve. All compounds must be detected in the MDL standard.

3. Internal standard calibration consists of analyzing six distinct levels of analyte and surrogate concentrations and producing response factors for each compound (six levels are required if second order regression fits are used). For medium/high level soils, methanol is added to all calibration standards. For low-level soils prepared according to EPA Method 5035, sodium bisulfate is added to all calibration standards.

4. The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of the analyte concentration.

5. When using an Archon (low/high) or OI 4551 (high) autosampler, blanks and standards are prepared and poured into 40-mL vials with Teflon-lined septa. For the high-level method, 5 mL is withdrawn from the vial and transferred to the sparge vessel along with the appropriate amount of the internal standard spiking solution. For the low-level method, the Archon transfers 5 mL of reagent water along with the appropriate amount of internal standard spiking solution to the 40-mL vial.

6. Purge and desorb according to Procedure 2.

7. Collect GC/MS data until the end of the GC run.

8. Empty and rinse the purging chamber at least twice with reagent water prior to loading another sample into the vessel, to minimize the possibility of carryover contamination.

9. Each level is analyzed as described above. Next, tabulate the area response of the characteristic ions (Table IV) against concentration for each analyte, surrogate standard, and internal standard and calculate relative response factors (RRF) for each compound (see Calculation section).

Frequency	Acceptance Criteria	Corrective Action
Initially and then when CCCs and/or SPCCs in the daily calibration standard fail criteria. SPCCs: chloromethane; 1,1-dichloroethane; $CHBr_3$, 1,1,2,2,-tetrachloroethane; chlorobenzene CCCs: 1,1-dichloroethene, $CHCl_3$, 1,2,-dichloropropane, toluene, ethylbenzene and vinyl chloride	1. Average RRF for each SPCC ≥ 0.1 except for 1,1,2,2,-tetrachloroethane and chlorobenzene (average RRF should be ≥ 0.3) 2. %RSD for each CCC ≤ 30%. 3. %RSD for non-CCCs ≤ 50%. 4. All compounds of interest must be detected in the MDL standard. 5. The relative retention times of the target compounds must agree within 0.06 relative retention time (RRT) units. The exception would be in the case of system maintenance.	1. – 3. Any target analyte with an %RSD of ≤15% should use the average RRF for quantitation. For any analyte in which the %RSD >15%, a first-degree linear regression can be used {provided that the correlation coefficient (CC) is ≥ 0.99}. A quadratic fit ** (using 6 stds) also may be used {provided the coefficient of determination (CD) is ≥0.99}. If the linear fit and quadratic fit pass the criteria for any given analyte, then use the line/curve with the smallest positive y-intercept. If the y-intercept quantifies to be greater than the LOQ, consult your supervisor immediately or recalibrate. If CC or CD is <0.99, recalibrate. Supervisory approval is required for exceptions to these guidelines. 4. If a compound is not detected in the MDL standard, then report to the level of the lowest standard detected. 5. Perform system maintenance and recalibrate.

** - Consult USEPA method 8000B for non-linear curve fitting techniques/guidelines

E. Continuing Calibration:

Continuing Calibration Verification (CCV) – The CCV is performed by analyzing a calibration standard in subsequent tune periods after an initial calibration. The concentration of the CCV should be varied between runs, correlating to levels of the initial calibration (use the middle 3 standards). The CCV is considered valid when the criteria listed below are met:

Frequency	Acceptance Criteria	Corrective Action
1. Every 12 hours. 2. Vary the standard level between 3 of the points of the initial calibration, excluding the low and high points (ie. 20ppb, 50 ppb, and 100ppb). SPCCs: chloromethane; 1,1-dichloroethane; $CHBr_3$, 1,1,2,2,-tetrachloroethane; chlorobenzene CCCs: 1,1-dichloroethene, $CHCl_3$, 1,2,-dichloropropane, toluene, ethylbenzene and vinyl chloride	1. Average RRF for each SPCC ≥0.1, except for 1,1,2,2,-PCA and chlorobenzene (avg. RRF should be ≥0.3) 2. %Drift for each CCC ≤20%. 3. %Drift for non-CCCs ≤50%. 4. The relative retention times (RRT) of the target compounds must agree within 0.06 RRT units. The exception would be in the case of system maintenance. 5. The extracted ion current profile (EICP) area for each internal standard must fall within the window of –50 % to +100 % from the average of the areas produced during the last initial calibration.	1. - 5. In the event that the continuing calibration verification (CCV) standard fails any of these criteria, sample analysis must be suspended and the CCV must be re-analyzed. If the re-analysis fails any of the criteria then adjustments are to be made to the analytical system to return it to its original condition, followed by the analyses of 2 consecutive CCVs at the same level that failed. If both CCVs pass the criteria, then sample analysis can continue. Otherwise, the system must be recalibrated and the samples reanalyzed, or the data can be qualified.

Analyze the method blank as described above for the initial calibration standards. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than the quantitation limit. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met. Also, all surrogate compound recoveries must meet the criteria listed in Table III.

Calibration Calculations:

1. Calculation of the relative response factor (RRF):

$$RRF=\frac{[A(x)\times C(is)]}{[A(is)\times C(x)]}$$

Where:

A(x) = Characteristic ion area for the compound being measured

A(is) = Characteristic ion area for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression equations:

$$\textit{1st Order (linear) regression :} \quad Y = Mx + B$$
$$\textit{2nd order (quadratic) regression :} \quad Y = Cx2 + Mx + B$$

Where:

x = Area(Std) /Area(Istd)

Y = Conc.(Std)/Conc.(Istd)

M = 1st degree slope

C = 2nd degree slope

B = Y-intercept

3. Percent relative standard deviation (%RSD) :

$$\%\ RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

4. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

C(c) = Measured concentration using selected quantification method

Qualitative Analysis:

Sample analysis for soil, solids, and nonaqueous matrices proceeds as described in the Procedure section. The aqueous matrix is replaced by the soil sample, which is prepared according to Analysis #0374 or Analysis #8389, 8390. For #0374 (low level) and 8389, 5 mL of reagent water with the internals and surrogate standards must be added to the sample prior to purging. The high level sample extract prepared by #8390 should be spiked with the appropriate amount of the 2500 µg/mL surrogate solution prior to removing an aliquot for analysis. Since the surrogate is already added for the high level (and medium level #0374), only internal standards need to be added at the time of analysis. A compound is identified by comparison of the following parameters with those of a standard of this target compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The compound relative retention time should compare within ±0.06 RRT units of the RRT of the standard.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The relative intensities of the characteristic ions should agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst discretion is used to determine compound identification. (Example: for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

5. The above criteria apply to hits greater than or equal to the LOQ. For hits between the MDL and the LOQ, both the above criteria and analyst discretion are used to determine compound identification.

6. The analyst must account for peaks that are greater than 10% relative intensity in the sample mass spectrum, but not present in the standard mass spectrum. Also, If a compound fails any of the criteria listed above but in the judgement of the mass spectral interpretation specialist is a correct identification, the identification is used and the quantitation of the peak is performed.

   The primary and secondary ions can be found in Table IV.

Quantitative Analysis:

Once a compound has been identified, quantitation of identified priority pollutant compounds is performed using the equations listed in the Calculations section of this procedure. The primary ions listed in Table IV are used for quantitation. A secondary ion may be used if there is interference with the primary ion. All calculations should report concentrations in values of µg/kg. Any analyte with a calculated concentration above the highest standard must be reanalyzed at a dilution that will bring the concentration in solution within the calibration curve. It is desirable to have the dilution fall within the top half of the calibration curve, but it is not required.

1. Low level

$$Concentration\ (\mu g / kg) = \frac{(Ax)\ (Is)}{(Ais)\ (RRF)\ (Ws)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Amount of internal standard added in nanograms

Ws = Weight of sample purged

RRF = Relative response factor from the initial calibration

2. Medium/high level

$$Concentration\ (\mu g / kg) = \frac{(Ax)\ (Is)\ (Vt)}{(Ais)\ (RRF)\ (Ws)\ (Vi)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Amount of internal standard added in nanograms

Vt = Volume of the total extract in microliters

Vi = Volume of the extract used for purging in microliters

Ws = Weight of sample extracted

RRF = Relative response factor from the initial calibration

Quality Assurance/Quality Control:

Each analysis batch must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD), a laboratory control sample/laboratory control sample duplicate (LCS/LCSD) or a duplicate (DUP). Additional QC samples may be required to meet project or state certification requirements. Every sample or QC analysis must contain internal standards and surrogate compounds.

The GC/MS system must be tuned to meet the criteria in Table II following BFB injection. The chromatographic conditions must be the same as those under which the samples will be analyzed except that the rate of temperature ramping may be increased and the initial temperature and column flow may be different. The BFB tune must be verified every 12 hours.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards • Added to every sample, standard, method blank or QC sample	1. Peak areas within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If re-injecting meets the criteria, report this analysis. 3. If this reanalysis still shows the same problem, report results from first analysis and qualify data with a comment.
Surrogates • Added to every sample, standard, method blank or QC sample	All % recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi-annual basis.	If non-compliant, check for calculation or preparation errors. If no errors are found, check system for problems and reanalyze. If this reanalysis still shows the same problem, report first analysis and qualify data with a comment.
Method Blank (MB) • Performed during each tune period after the initial calibration or CCV (minimum of 1 MB per 20 samples)	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. Quantitative results for all target compounds must be less than the reporting limit for the associated samples.	1. – 2. Inspect system for possible problems and reanalyze. 3. If the MB contains target analytes and the associated samples do not, then no corrective action is required. If the target compounds in the MB are also in the associated samples, then they should be reanalyzed after a clean MB is obtained (certain projects may allow some exceptions for common laboratory contaminants like methylene chloride and acetone up to 5X the LOQ)
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) • LCS analyzed with each batch of ≤ 20 samples • LCSD analyzed if MSD unavailable • See Tables V and VI for preparation info.	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All % recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi-annual basis.	1. - 2. If non-compliant, check for calculation or preparation errors. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD re-analysis still fails, perform appropriate system maintenance and restart the tune period. Only with an LCS % recovery failing high (for the requested target compounds) with targets non-detected in the sample, can the results be reported. Otherwise, the sample must be analyzed with a compliant LCS.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) • MS analyzed with each batch of ≤ 20 samples (if sufficient sample volume available) • See Tables V and VI for preparation info.	1. % Recoveries should fall within statistically derived QC limits, which are reviewed and updated on a semi-annual basis 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries and/or RPDs outside of QC limits, consult the supervisor.

The method blank must meet all the above criteria for internal standard recoveries and surrogate recoveries. In addition, the method blank may not contain any target compound above the quantitation limit. All method blanks must meet these criteria; otherwise, the system is considered out of control and corrective action must be taken.

If sufficient sample is not available to perform MS/MSD, LCS/LCSD are prepared and analyzed and must meet the above-mentioned criteria.

NOTE: Prior to release from the analytical laboratory, a data review specialist, with respect to quality assurance and data interpretation reviews all data. The data review specialist will return data to the analyst for correction as necessary. The specialist will also report the results of the review and corrections on a form to be kept with the raw data. Any noncompliant data that cannot be corrected will be referred to the group leader for further action. Refer to SOP-MS-012.

Sample Analysis:

The sample is analyzed using the same instrumental conditions as the standard (whether ICAL or CCV), tune and method blank. If the QA criteria are satisfied and no target compounds are detected at concentrations above the calibration range, the results can be reported. To avoid possible matrix effects, sample carryover and re-analyses, an initial dilution may be performed if:

1. Prescreening indicates a high volatile organic content in the sample

2. Historical data (or lack thereof) and/or sample appearance indicate a need for dilution

If target compounds are detected in the sample at concentrations above the calibration range, a dilution must be performed (see SOP-MS-007 for information on when cleaning blanks must be run). See Section 7.5.6 in method SW-846 8260B for recommended dilution procedures.

QC Calculations:

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

Relative percent difference (RPD)

$$RPD = \frac{MSR - MSRD}{(1/2)(MSR + MSRD)} \times 100$$

Where:

MSR = Matrix spike measured concentration

MSDR = Matrix spike duplicate measured concentration

Revision Log:

Ver. #	Effective Date	Change
00	10/15/96	Previous Issue
01	01/05/98	Major changes: • Updated entire method to comply with 8260B
02	04/14/98	Major changes: • 8260B Updates • Added analysis numbers

Ver. #	Effective Date	Change
03	03/25/99	Major changes: • Update information on correlation coefficient and coefficient of determination • Update requirement on checking areas in calibration check standard • Update LCS requirement • Added analysis numbers 2304, 2305, 2307, 2308, 2310, 8795
04	08/28/00	Major changes are as follows: • Cross Reference – Section added • Procedure – Added requirement to add preservative to calibration standards, added %RSD and regression calculations • Sample Analysis – Added % recovery calculation • Incorporated Procedural Amendments #1 and 2
05	08/08/02	Major changes are as follows: • Updated the following sections: Cross Reference, Apparatus, Standards, Procedure, Data Analysis, and Quality Assurance • Updated Table 2 with current TSC Sheet • Added Table IV • Deleted scans 2305, 2307, 2309, and 8795 • Added scan 4514 • Added onto Table I • Incorporated Procedural Amendment #1 • Updated Addendum section • Reformatted to Level 3

Ver. #	Effective Date	Change
06	11/04/03	Major changes are as follows: • Revised Tables I-A thru I-L • Reformatted QC requirements in the tabular form • Revised References and Cross References • Major rewrite and reorganized
07	JUL 14 2005	Major changes are as follows: • Table V: Added updated TSC sheet • Table VI : Added updated TSC sheet

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Prepared by: _____ Date: 6/30/05
Senior Chemist, Group Leader

Approved by: _____ Date: 6/30/05
GC/MS Volatiles Management

Approved by: _____ Date: 6/30/05
GC/MS Volatiles Management

Approved by: _____ Date: 6/30/05
Quality Assurance

Table I-A
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #2304

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Methyl *t*-Butyl Ether	5
Benzene	5
Toluene	5
Ethylbenzene	5
Total Xylenes	5
Isopropylbenzene	5
Naphthalene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-B
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #2308

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Methyl *t*-Butyl Ether	5
Benzene	5
Toluene	5
Ethylbenzene	5
Total Xylenes	5
Isopropylbenzene	5
Naphthalene	5
1,2-Dichloroethane	5
1,2-Dibromoethane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-C
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #2310

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Methyl *t*-Butyl Ether	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-D
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #4514

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Methyl *t*-Butyl Ether	5
Benzene	5
Toluene	5
Ethylbenzene	5
m+p-Xylene	5
o-Xylene	5
Isopropylbenzene	5
n-Propylbenzene	5
1,3,5-Trimethylbenzene	5
tert-Butylbenzene	5
1,2,4-Trimethylbenzene	5
sec-Butylbenzene	5
p-Isopropyltoluene	5
n-Butylbenzene	5
Naphthalene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-E
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #5441, 5442

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Benzene	5
Bromobenzene	5
Bromochloromethane	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
n-Butylbenzene	5
sec-Butylbenzene	5
tert-Butylbenzene	5
Carbon tetrachloride	5
Chlorobenzene	5
Chlorodibromomethane	5
Chloroethane	5
Chloroform	5
Chloromethane	5
2-Chlorotoluene	5
4-Chlorotoluene	5
1,2-Dibromo-3-chloropropane	5
1,2-Dibromoethane	5
Dibromomethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
Dichlorodifluoromethane	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
cis-1,2-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
1,3-Dichloropropane	5
2,2-Dichloropropane	5
1,1-Dichloropropene	5
Ethylbenzene	5
Hexachlorobutadiene	5
Isopropylbenzene	5
p-Isopropyltoluene	5
Methylene chloride	5
Naphthalene	5

Table I-E
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #5441, 5442

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
n-Propylbenzene	5
Styrene	5
1,1,1,2-Tetrachloroethane	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,2,3-Trichlorobenzene	5
1,2,4-Trichlorobenzene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
1,2,3-Trichloropropane	5
1,2,4-Trimethylbenzene	5
1,3,5-Trimethylbenzene	5
Vinyl chloride	5
m+*p*-Xylene	5
o-Xylene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-F
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #6292

Volatiles	Practical Quantitation Limits Soils/Solids (ug/kg)
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
1,1-Dichloroethene	5
Acetone	20
Carbon Disulfide	5
Methylene Chloride	5
1,1-Dichloroethane	5
trans-1,2-Dichloroethene	5
cis-1,2-Dichloroethene	5
2-Butanone	10
Chloroform	5
1,2-Dichloroethane	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Benzene	5
Trichloroethene	5
1,2-Dichloropropane	5
Bromodichloromethane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
Dibromochloromethane	5
Bromoform	5
4-Methyl-2-pentanone	10
Toluene	5
Tetrachloroethene	5
2-Hexanone	10
Chlorobenzene	5
Ethylbenzene	5
Xylene (total)	5
Styrene	5
1,1,2,2-Tetrachloroethane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-G
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #7360

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Methyl *t*-Butyl Ether	5
Benzene	5
Toluene	5
Ethylbenzene	5
Xylene (Total)	5

Table I-H
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #7361

Volatiles	Practical Quantitation Limits Water/Wastewater (µg/kg)
Methyl *t*-Butyl Ether	5
Benzene	5
Toluene	5
Ethylbenzene	5
Xylene (Total)	5
1,2-Dichloroethane	5
Di-Isopropyl Ether	5
Ethyl *t*-Butyl Ether	5
t-Amyl Methyl Ether	5
t-Butyl Alcohol	100
1,2-Dibromoethane	5

Table I-I
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #7584

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluoromethane	5
Acrolein	100
1,1-Dichloroethene	5
Methylene Chloride	5
Acrylonitrile	50
trans-1,2-Dichloroethene	5
1,1-Dichloroethane	5
cis-1,2-Dichloroethene	5
Chloroform	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Benzene	5
1,2-Dichloroethane	5
Trichloroethene	5
1,2-Dichloropropane	5
Bromodichloromethane	5
2-chloroethyl vinyl ether	10
cis-1,3-Dichloropropene	5
Toluene	5
trans-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
Tetrachloroethene	5
Dibromochloromethane	5
Chlorobenzene	5
Ethylbenzene	5
Xylene (total)	5
Bromoform	5
1,1,2,2-Tetrachloroethane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-J
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #7720

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
Dichlorodifluoromethane	5
Chloromethane	5
Vinyl Chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluoromethane	5
Acrolein	100
1,1-Dichloroethene	5
Acetone	20
Methyl Iodide	5
Carbon Disulfide	5
Acetonitrile	100
Allyl Chloride	5
Methylene Chloride	5
Acrylonitrile	50
trans-1,2-Dichloroethene	5
1,1-Dichloroethane	5
Vinyl Acetate	10
2-Chloro-1,3-butadiene	5
cis-1,2-Dichloroethene	5
2-Butanone	10
Propionitrile	100
Methacrylonitrile	50
Chloroform	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Isobutyl Alcohol	250
Benzene	5
1,2-Dichloroethane	5
Trichloroethene	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-K
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis #7721

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
1,4-Dioxane	250
Bromodichloromethane	5
cis-1,3-Dichloropropene	5
4-Methyl-2-pentanone	10
Toluene	5
trans-1,3-Dichloropropene	5
Ethyl Methacrylate	5
1,1,2-Trichloroethane	5
Tetrachloroethene	5
2-Hexanone	10
Dibromochloromethane	5
1,2-Dibromoethane	5
Chlorobenzene	5
1,1,1,2-Tetrachloroethane	5
Ethylbenzene	5
Xylene (total)	5
Styrene	5
Bromoform	5
1,1,2,2-Tetrachloroethane	5
1,2,3-Trichloropropane	5
trans-1,4-Dichloro-2-butene	50
Pentachloroethane	10
1,2-Dibromo-3-chloropropane	5

*Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

Table I-L
Practical Quantitation Limits (PQL) for Volatile Organics*
Analysis # 6373

Volatiles	Practical Quantitation Limits Soils/Solids (µg/kg)
1,1,1,2-Tetrachloroethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
n-Butanol	250
Methyl Methacrylate	5
Dichlorodifluoromethane	5
1,2-Dichloroethane	5
Bromochloromethane	5
2-Nitropropane	10
2-Chloroethyl Vinyl Ether	10
Trichlorofluoromethane	5
Freon 113	10
Acrolein	100
Acrylonitrile	20
Isopropylbenzene	5
Methyl Tertiary Butyl Ether	5
t-Butyl alcohol	100
1,2-Dibromoethane	5
1,4-Dioxane	250
1,2,3-Trichloropropane	5
Chlorodifluoromethane	10
1,2,4,5-Tetramethylbenzene	5
4-Ethyltoluene	5
1,2-Dichloroethene (Total)	5
Acetone	20
Carbon Disulfide	5
2-Butanone	10
4-Methyl-2-pentanone	10
2-Hexanone	10
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Naphthalene	5
Vinyl Acetate	10
n-Heptane	5
n-Hexane	5

***Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable.**

Table II
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Table III
Surrogate Spike Recovery Limits for Soils and Solids Samples

Surrogate Compound	Soils/Solids
4-Bromofluorobenzene	70-128
Dibromofluoromethane	70-129
Toluene-d8	70-130
1,2-Dichloroethane-d4	70-121

Table IV
Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
trans-1,2-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77, 110
trans-1,3-Dichloropropene	75	77, 110
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzen-d4	152	115

Table V

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B

Date:_____
Instrument:_____

VOA1= 1:5 dilution of VCS#1B, VCS#2B, and VCS#4C
VOA2= 1:5 dilution of VCS#2B
VOA3= 1:5 dilution of VCS#3B and Vacrolein
VOA6= 1:5 dilution of VCS#6
2CEVE= 1:5 dilution of VCS#1B-2CEVE

Stock mix name	VOA1 _____ 2CEVE _____	VOA3 _____	VOA2 _____	VOA6 _____ EE _____	EOH _____	CYC _____	Restek Gases (2000 ppm) Lt#_____	IH826SS 250 ppm @ _____ 2500 ppm 8260 SS $ Lt#_____	Flask mL	MeOH ml B&J Brand Lt_____
300 ppb std	15 µL	6 µL		15µL	60 µL	30 µL	7.5 µL	60 µL @/ 6 µL $	50	1
100 ppb std	5 µL	2 µL		5 µL	20 µL	10µL	2.5 µL	20 µL @/ 2 µL $	50	1
50 ppb std	5 µL	2 µL		5 µL	20 µL	10µL	2.5 µL	20ul @/ 2ul $	100	2
20 ppb std	4 µL	1.6 µL	4 µL	4 µL	32 µL	16µL	2.0 µL	16 µL@/1.6 $	200	4
10 ppb std	2 µL	0.8 µL	2 µL	2 µL	16 µL	8µL	1.0 µL	8 µL@/0.8 $	200	4
4 ppb std	4 µL	1.6 µL	12 µL	4 µL	40 µL	32µL	2.0 µL	16 µL@/1.6 $	1000 *	20
								@ µL of 250 ppm std used $ µL of 2500 ppm std used		
1 ppb std	* Aliquot 12.5 mL of 1000 mL flask into 50 mL flask									

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb
Benzene	CS#1B	5000	300	100	50	20	10	4	1
Bromobenzene		5000	300	100	50	20	10	4	1
Bromodichloromethane		5000	300	100	50	20	10	4	1
Bromoform		5000	300	100	50	20	10	4	1
n-Butylbenzene		5000	300	100	50	20	10	4	1
sec-Butylbenzene		5000	300	100	50	20	10	4	1
tert-Butylbenzene		5000	300	100	50	20	10	4	1
Carbon Tetrachloride		5000	300	100	50	20	10	4	1
Chlorobenzene		5000	300	100	50	20	10	4	1
Chloroform		5000	300	100	50	20	10	4	1
2-Chlorotoluene		5000	300	100	50	20	10	4	1
4-Chlorotoluene		5000	300	100	50	20	10	4	1
Dibromochloromethane		5000	300	100	50	20	10	4	1
1,2-Dibromo-3-chloropropane		5000	300	100	50	20	10	4	1
1,2-Dibromoethane (EDB)		5000	300	100	50	20	10	4	1
Dibromomethane		5000	300	100	50	20	10	4	1
1,2-Dichlorobenzene		5000	300	100	50	20	10	4	1
1,3-Dichlorobenzene		5000	300	100	50	20	10	4	1
1,4-Dichlorobenzene		5000	300	100	50	20	10	4	1

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Table V – Continued

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb
1,1-Dichloroethane	CS#1B	5000	300	100	50	20	10	4	1
1,2-Dichloroethane		5000	300	100	50	20	10	4	1
1,1-Dichloroethene		5000	300	100	50	20	10	4	1
cis-1,2-Dichloroethene		5000	300	100	50	20	10	4	1
trans-1,2-Dichloroethene		5000	300	100	50	20	10	4	1
1,2-Dichloropropane		5000	300	100	50	20	10	4	1
1,3-Dichloropropane		5000	300	100	50	20	10	4	1
2,2-Dichloropropane		5000	300	100	50	20	10	4	1
1,1-Dichloropropene		5000	300	100	50	20	10	4	1
cis-1,3-Dichloropropene		5000	300	100	50	20	10	4	1
trans-1,3-Dichloropropene		5000	300	100	50	20	10	4	1
Ethylbenzene		5000	300	100	50	20	10	4	1
Hexachlorobutadiene		5000	300	100	50	20	10	4	1
Isopropylbenzene (Cumene)		5000	300	100	50	20	10	4	1
p-Isopropyltoluene		5000	300	100	50	20	10	4	1
Methylene Chloride		5000	300	100	50	20	10	4	1
Naphthalene		5000	300	100	50	20	10	4	1
n-Propylbenzene		5000	300	100	50	20	10	4	1
Styrene		5000	300	100	50	20	10	4	1
1,1,1,2-Tetrachloroethane		5000	300	100	50	20	10	4	1
1,1,2,2-Tetrachloroethane		5000	300	100	50	20	10	4	1
Tetrachloroethene		5000	300	100	50	20	10	4	1
Toluene		5000	300	100	50	20	10	4	1
1,2,3-Trichlorobenzene		5000	300	100	50	20	10	4	1
1,2,4-Trichlorobenzene		5000	300	100	50	20	10	4	1
1,1,1-Trichloroethane		5000	300	100	50	20	10	4	1
1,1,2-Trichloroethane		5000	300	100	50	20	10	4	1
Trichloroethene		5000	300	100	50	20	10	4	1
1,2,3-Trichloropropane		5000	300	100	50	20	10	4	1
1,2,4-Trimethylbenzene		5000	300	100	50	20	10	4	1
1,3,5-Trimethylbenzene		5000	300	100	50	20	10	4	1
m-Xylene		5000	300	100	50	20	10	4	1
o-Xylene		5000	300	100	50	20	10	4	1
p-Xylene		5000	300	100	50	20	10	4	1
2-Chloroethyl Vinyl Ether	2CEVE	5000	300	100	50	20	10	4	1
Bromomethane	Gas	2000	300	100	50	20	10	4	1
Chloroethane	mix	2000	300	100	50	20	10	4	1
Chloromethane		2000	300	100	50	20	10	4	1
Dichlorodifluoromethane		2000	300	100	50	20	10	4	1
Trichlorofluoromethane		2000	300	100	50	20	10	4	1
Vinyl Chloride		2000	300	100	50	20	10	4	1

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Table V – Continued

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb
Methacrylonitrile	CS#2B	12500	750	250	125	100	50	40	10
Propionitrile		25000	1500	500	250	200	100	80	20
trans-1,4-Dichloro-2-Butene		12500	750	250	125	100	50	40	10
t-Butyl Alcohol		25000	1500	500	250	200	100	80	20
2-Propanol		25000	1500	500	250	200	100	80	20
Isobutyl Alcohol		62500	3750	1250	625	500	250	200	50
n-Butanol		125000	7500	2500	1250	1000	500	400	100
1,4-Dioxane		62500	3750	1250	625	500	250	200	50
2-Butanone	CS#3B	25000	600	200	100	40	20	8	2
2-Hexanone		25000	600	200	100	40	20	8	2
4-Methyl-2-Pentanone		25000	600	200	100	40	20	8	2
Acetone		25000	600	200	100	40	20	8	2
Acrylonitrile		12500	300	100	50	20	10	4	1
2-Nitropropane		25000	600	200	100	40	20	8	2
Tetrahydrofuran		25000	600	200	100	40	20	8	2
Methyl-t-butyl Ether	CS#4C	5000	300	100	50	20	10	4	1
Ethyl Methacrylate		5000	300	100	50	20	10	4	1
Methyl Methacrylate		5000	300	100	50	20	10	4	1
Freon 113		5000	300	100	50	20	10	4	1
Hexane		5000	300	100	50	20	10	4	1
Heptane		5000	300	100	50	20	10	4	1
Cyclohexane		5000	300	100	50	20	10	4	1
Benzyl Chloride		5000	300	100	50	20	10	4	1
Methyl Iodide		5000	300	100	50	20	10	4	1
Carbon Disulfide		5000	300	100	50	20	10	4	1
2-Chloro-1,3-Butadiene		5000	300	100	50	20	10	4	1
di-Isopropyl Ether		5000	300	100	50	20	10	4	1
tert-Amyl Methyl Ether		5000	300	100	50	20	10	4	1
Ethyl-t-butyl Ether		5000	300	100	50	20	10	4	1
Pentachloroethane	CS#6	5000	300	100	50	20	10	4	1
Allyl Chloride		5000	300	100	50	20	10	4	1
Bromochloromethane		5000	300	100	50	20	10	4	1
Methyl Acetate		5000	300	100	50	20	10	4	1
Methylcyclohexane		5000	300	100	50	20	10	4	1
2-Methylnaphthalene		5000	300	100	50	20	10	4	1
1,2,3-Trimethylbenzene		5000	300	100	50	20	10	4	1
1,2-Diethylbenzene		5000	300	100	50	20	10	4	1
1,3-Diethylbenzene		5000	300	100	50	20	10	4	1
1,4-Diethylbenzene		5000	300	100	50	20	10	4	1

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Table V – Continued

Theoretical Standard Concentrations
Initial Calibration for Large Curve
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B

Compound name	std mix	Stock ppm	300 ppb	100 ppb	50 ppb	20 ppb	10 ppb	4 ppb	1 ppb
Ethyl Ether	EE	1000	300	100	50	20	10	4	1
n-Pentane	n-PEN	1000	300	100	50	20	10	4	1
1,2,4,5-Tetramethylbenzene	CVM	1000	300	100	50	20	10	4	1
Chlorodifluoromethane		2000	600	200	100	40	20	8	2
4-Ethyltoluene		1000	300	100	50	20	10	4	1
1,4-Diethylbenzene		1000	300	100	50	20	10	4	1
Acrolein	VACR	125000	3000	1000	500	200	100	40	10
Cyclohexanone	CYC	6250	3750	1250	625	500	250	200	50
Ethanol	EOH	12500	15000	5000	2500	2000	1000	500	125

ppb of analytical standard = (stock ppm)(µL stock) / flask mL

Analyst:_____
Date:_____

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Table VI

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B
Low Soil and NJ MeOH Prep

QVOA6 = 1: 25 QCS#6 Date:_____
QARC = 1: 25 QCS#1B2CEVE, QACR stock Instrument:_____
QVOA1= 1:25 QCS#1B, QCS#2B, QCS3B, QCS#4C QGASES=1:50 Restek 502.2 "Q" Gas mix
Qn-pentane = 40ul of n-pentane lot#_____ to 960ul B&J Brand MEOH lot# _____
QBUT = 40ul of 1,3-Butadiene lot#_____ to 960ul B&J Brand MEOH lot# _____

Stock mix Name	QVOA1 / QARC / QBUT	QVOA6 / QEE / QCYC	QEOH	8260 SS 2500 ppm Lot#____	QGASES / Qn-pentane	Final Volume	MeOH Lot#	Used
20 ppb	2.5 µL	2.5 µL	5.0 µL	0.1 ul	2.5 µL	5 mL Syringe	.1 mL	
20 ppb	21.5 µL	21.5 µL	43.0 µL	-	21.5 µL	43 mL Vial	-	
20 ppb	25.0 µL	25.0 µL	50.0 µL	1.0 ul	25.0 µL	50 mL Flask	1 mL	

Compound name	std mix	Stock ppm	20 ppb
Benzene	QCS#1B	1000	20
Bromobenzene		1000	20
Bromodichloromethane		1000	20
Bromoform		1000	20
n-Butylbenzene		1000	20
sec-Butylbenzene		1000	20
tert-Butylbenzene		1000	20
Carbon Tetrachloride		1000	20
Chlorobenzene		1000	20
Chloroform		1000	20
2-Chlorotoluene		1000	20
4-Chlorotoluene		1000	20
Dibromochloromethane		1000	20
1,2-Dibromo-3-chloropropane		1000	20
1,2-Dibromoethane (EDB)		1000	20
Dibromomethane		1000	20
1,2-Dichlorobenzene		1000	20
1,3-Dichlorobenzene		1000	20
1,4-Dichlorobenzene		1000	20
1,1-Dichloroethane		1000	20
1,2-Dichloroethane		1000	20
1,1-Dichloroethene		1000	20
cis-1,2-Dichloroethene		1000	20
trans-1,2-Dichloroethene		1000	20
1,2-Dichloropropane		1000	20
1,3-Dichloropropane		1000	20
2,2-Dichloropropane		1000	20
1,1-Dichloropropene		1000	20
cis-1,3-Dichloropropene		1000	20
trans-1,3-Dichloropropene		1000	20
Ethylbenzene		1000	20
Hexachlorobutadiene		1000	20

Table VI – Continued

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B
Low Soil and NJ MeOH Prep

Compound name	std mix	Stock ppm	20 ppb
p-Isopropyltoluene	QCS#1B	1000	20
Methylene Chloride		1000	20
Isopropylbenzene (Cumene)		1000	20
Naphthalene		1000	20
n-Propylbenzene		1000	20
Styrene		1000	20
1,1,1,2-Tetrachloroethane		1000	20
1,1,2,2-Tetrachloroethane		1000	20
Tetrachloroethene		1000	20
Toluene		1000	20
1,2,3-Trichlorobenzene		1000	20
1,2,4-Trichlorobenzene		1000	20
1,1,1-Trichloroethane		1000	20
1,1,2-Trichloroethane		1000	20
Trichloroethene		1000	20
1,2,3-Trichloropropane		1000	20
1,2,4-Trimethybenzene		1000	20
1,3,5-Trimethylbenzene		1000	20
m-Xylene		1000	20
o-Xylene		1000	20
p-Xylene		1000	20
Bromomethane	QGas	2000	20
Chloroethane	mix	2000	20
Chloromethane		2000	20
Dichlorodifluoromethane		2000	20
Trichlorofluoromethane		2000	20
Vinyl Chloride		2000	20
Methacrylonitrile	QCS#2B	7500	150
Propionitrile		7500	150
trans-1,4-Dichloro-2-Butene		5000	100
t-Butyl Alcohol		10000	200
2-Propanol		7500	150
Isobutyl Alcohol		25000	500
n-Butanol		50000	1000
1,4-Dioxane		25000	500
2-Butanone	QCS#3'3	7500	150
2-Hexanone		5000	100
4-Methyl-2-Pentanone		5000	100
Acetone		7500	150
Acrylonitrile		5000	100
2-Nitropropane		1000	20
Tetrahydrofuran		5000	100

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Table VI – Continued

Theoretical Standard Concentrations
Quality Control
Purchased Standards
HP Capillary Column
EPA SW846 Method 8260A/B
Low Soil and NJ MeOH Prep

Compound name	Std mix	Stock ppm	20 ppb
Methyl-t-butyl Ether	QCS#4C	1000	20
Ethyl Methacrylate		1000	20
Methyl Methacrylate		1000	20
Freon 113		1000	20
Hexane		1000	20
Heptane		1000	20
Cyclohexane		1000	20
Benzyl Chloride		1000	20
Methyl Iodide		1000	20
Carbon Disulfide		1000	20
2-Chloro-1,3-Butadiene		1000	20
di-Isopropyl Ether		1000	20
tert-Amyl Methyl Ether		1000	20
Ethyl-t-butyl Ether		1000	20
Pentachloroethane	QCS#6	1000	20
Allyl Chloride		1000	20
Bromochloromethane		1000	20
Methyl Acetate		1000	20
Methylcyclohexane		1000	20
2-Methylnaphthalene		1000	20
1,2,3-Trimethylbenzene		1000	20
1,2-Diethylbenzene		1000	20
1,3-Diethylbenzene		1000	20
1,4-Diethylbenzene		1000	20
Acrolein	QACR	7500	150
2- Chloroethyl Vinyl Ether	QCS#1B 2CEVE	1000	20
Cyclohexanone	QCYC	1000	500
Ethyl Ether	QEE	40	20
n-Pentane	Qn-PEN	40	20
1,3-Butadiene	QBUT	40	20
Ethanol	QEOH	1000	1000

ppb of analytical standard = (stock ppm)(µl stock) / final volume

Analyst:_____
Date:_____

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Analysis # 2304, 2308, 2310, 4514, 5441, 5442, 6292, 6373, 7360, 7361, 7584, 7720, 7721
Revision 07 PA #1
Procedure Effective Date: 07/14/05
PA Effective Date:
Page 1 of 1 APR 2 4 2006

Procedural Amendment #1

Procedure Title:

Determination of Volatile Target Compounds by Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS) in Soils and Solids by Method 8260B

Reasons for addition(s) or change(s):

Sample Handling section was left blank in previous review

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Sample Handling: *Add the following sentence:*

The samples should be stored in a refrigerator between 2° and 6°C. All samples must be analyzed within 14 days of collection.

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Prepared by: Kenneth L Boley, Jr. Date: 4/7/06
Senior Chemist

Approved by: [signature] Date: 4/20/06
GC/MS Volatiles Management

Approved by: [signature] Date: 4/21/06
GC/MS Volatiles Management

Approved by: Barbara F. Reedy Date: 4/24/06
Quality Assurance

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Analysis #5709, 5708
Revision 08
Supersedes Date: 10/03/05
Effective Date: AUG 16 2006

Sample Preparation of Sediments, Sludges, and Soils for Analysis of Metals by Atomic Absorption (5709) or Inductively Coupled Plasma Atomic Emission Spectrometry (5708)

Approvals:

Prepared by: Debra A. Bryan — Specialist, Group Leader — Date: 7-31-06

Approved by: Robert Strachan — Metals Management — Date: 8.1.06

Approved by: Elaine Stoltzfus — Quality Assurance — Date: 8/2/06

Revision Log:

Ver. #	Effective Date	Change
00	02/22/96	Previous Issue
01	01/15/98	Major changes are as follows: • Reference updated from Revision 1 to Revision 2. • Reference Modifications section was added. • Scope was revised to conform with Revision 2. • Antimony analysis by graphite furnace was removed as it will no longer be performed using this digestion. • Basic Principles was revised for clarification. • Personnel Training and Qualifications section was added. • Procedure was updated to conform with Revision 2
02	02/12/98	Major changes are as follows: • Reference - Added environmental lead program requirements • Environmental Lead Program QA Requirements - Expanded proficiency demonstration paragraph to match updated HUD training/proficiency/documentation requirements
03	07/27/00	Major changes are as follows: • Reference – Removed environmental Lead Program Requirement as it is not applicable • Cross reference – Added section • Apparatus and Reagents - Procedure – Polypropylene vessels and covers replace beakers and watchglasses. Block digestor replaces hotplates. • Environmental Lead Programs Requirement – Removed section as it is not applicable

Ver. #	Effective Date	Change
04	05/17/01	Major changes are as follows: • Incorporated Procedural Amendment #1 • Procedure – 1. Clarification on spiking regulations • Cross Reference – Added SOP-IO-005 and SOP-IO-012 Added Calculation section
05	12/26/02	Major Changes are as follows: • Deleted the third paragraph in Procedure section. This is a duplication of number three of Procedure section.
06	08/14/03	Major changes are as follows: • Procedure – changed the acceptable weight range • Reformatted to Level 3
07	10/03/05	Major changes are as follows: • Incorporated Procedural Amendment #1
08	AUG 1 6 2006	Major changes are as follows: • Updated Cross Reference section • Updated Reference Modifications section • Updated Apparatus and Equipment section • Procedure section – added item 6.

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Reference:

1. Method 3050B, *Test Methods for Evaluating Solid Waste,* SW-846, Third Edition, Revision 2, Part I, Chapter 3, USEPA, Office of Solid Waste and Emergency Response, Washington DC 20460, (December 1996).

2. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
LOM-SOP-ES-207	Establishing Control Limits
SOP-IO-001	Preservation, Storage Conditions, and Holding Times for Inorganic Samples
SOP-IO-007	Preparation of Standards and Solutions
SOP-IO-007, Section H	Prep Room Solutions (Solids)
SOP-IO-011	Inorganic Analysis Safety and Waste Handling Procedures
SOP-IO-012	Calculations Used by the Inorganics Group
SOP-IO-014	Quality Control Procedure for ICP

Purpose:

This acid digestion procedure is used to prepare sediment, sludge, and soil samples for analysis of metals by flame atomic absorption (FLAA) or inductively coupled plasma atomic emission spectrometry (ICP-AES) following SW-846 protocol.

Scope:

This method is not a **total** digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available." By design, elements bound in silicate structures are not normally dissolved by this procedure, as they are not usually mobile in the environment.

Basic Principles:

A representative 1-g as-received sample is digested with repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). Hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed. The resultant digestate is filtered and diluted to a final volume of 100 mL.

Reference Modifications:

When it is necessary to filter a sample after digesting, instead of diluting to 100 mL with deionized water and then filtering to remove particulates, for improved accuracy, samples are filtered through Whatman No. 41 filter paper or equivalent into a polypropylene digestion container and then made up to volume.

Interferences:

Not applicable to this method.

Safety Precautions and Waste Handling:

Refer to SOP-IO-011.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications:

Training and proof of proficiency for this procedure includes but is not limited to the following:

1. Review and understanding of this procedure

2. Trainee observing trained analyst performing procedure

3. Trainer observing trainee performing procedure

4. Review of trainee's data by trainer

5. Acceptable performance on quad studies

6. Documentation of critical steps in training process

Sample Handling:

For sample preservation, storage conditions, and holding times, see SOP-IO-001.

Apparatus and Equipment:

1. Polypropylene containers [digestion vessels]

2. Polypropylene covers [digestion vessel covers]

3. Whatman No. 41 filter paper or equivalent

4. Funnels

5. Environmental Express HotBlock [block digestor], adjustable and capable of maintaining a temperature of 95°C

6. Balance capable of reading 0.01 g

Reagents and Standards:

For reagent preparation, shelf life, and storage conditions, see SOP-IO-007.

1. Nitric acid, HNO_3, 70.0% to 71.0%; Baker Instra-Analyzed reagent, 1.428 g/mL or equivalent

2. Nitric acid (1:1) – Add 500 mL of HNO_3 to 500 mL of deionized water

3. Hydrogen peroxide, 30% H_2O_2, Fisher, Certified ACS or equivalent

4. Hydrochloric acid, HCl, 36.5% to 38.0%; Baker Instra-Analyzed reagent, 1.194 g/mL or equivalent

NOTE: As long as the correct ratios are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Procedure:

For sample preservation, storage conditions, and holding times, see SOP-IO-001.

Turn block digestor on and allow block to reach the Control Point setting that provides 95°C sample temperature[1].

1. Weigh 1 g (1.00 to 1.05 g to the nearest 0.01 g) of a thoroughly homogenized, as-received sample into a polypropylene digestion vessel (for sample batch spiking procedure see SOP-IO-007H and for sample batch quality control requirements see SOP-IO-014). Add 7 Corning PYREX 5-mm glass beads to the blank polypropylene digestion vessel. Add 10 mL of (1:1) HNO_3, swirl to mix, and cover with a polypropylene cover. Place sample vessel in block digestor. Heat [reflux] the sample at 95° ± 5°C for 10 to 15 minutes without boiling. Remove vessel from digestion block and allow sample to cool somewhat. Add 5 mL of concentrated HNO_3. Replace

cover, return vessel to digestion block and heat [reflux] for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this last step (addition of 5 mL of HNO_3) over and over until **no** brown fumes are given off by the sample indicating the complete reaction with HNO_3. Add the same amount of HNO_3 to the entire digestion batch. With cover on, heat at 95° ± 5°C without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times (add deionized water if required).

2. Remove vessel from digestion block and allow sample to cool somewhat. Add 2 mL of deionized water and 3 mL of 30% H_2O_2. With cover on, return vessel to digestion block and heat until effervescence subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

3. Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

   NOTE: Do not add more than a total of 10 mL 30% H_2O_2.

4. With cover on, continue heating the acid-peroxide digestate at 95° ± 5°C without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times (add deionized water if required). Remove sample vessel from digestion block and allow to cool somewhat.

5. Add 10 mL of HCl. With the cover on, return vessel to digestion block and heat [reflux] at 95° ± 5°C for 15 minutes. Remove sample vessel from digestion block.

6. When necessary, samples are to be filtered. Filter through Whatman No. 41 filter paper into a polypropylene container. Wash sample vessel, residue, and paper thoroughly with deionized water. Adjust volume to the mark with

deionized water and mix, seal vessel with a screw cap. The sample is now ready for analysis. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: Use approximately 5 g for soil, sediment, or sludge samples of watery consistency [slurries]. When special limits of quantitation are required by the client, use more sample weight.

NOTE: For paint chip samples, use approximately 0.5 g. If brown fumes are evolved from paint chip samples during digestion, perform only two 5 mL HNO_3 additions with 30 minute refluxing each; add the same amount of HNO_3 to the entire batch. Proceed with digestion.

NOTE: When wipes are digested by this method, one blank media each should be used for the batch preparation blank, the laboratory control sample [LCS], and the laboratory control sample duplicate [LCSD]. Spike the LCS and LCSD with CLP spike solutions. Digest wipes in their own batch. Using deionized water, rinse any particulate matter from the wipe container into the beaker containing the wipe sample before digesting. If brown fumes are evolved during wipe sample digestion, perform only two 5 mL HNO_3 additions with 30-minute refluxing each; add the same amount of HNO_3 to the entire batch. Proceed with digestion.

[1] **NOTE:** The block temperature is different than the temperature of the liquid being digested.

Turn block digestor on by pressing rocker switch located on the right rear panel. Wait about 8 seconds until controller display indicates current block temperature. Press Up Arrow key. The display will show Control Point temperature. Confirm Control Point temperature is set to the block temperature that provides 95°C. See Control Point label on HotBlock or HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting as instructed below.

Control Point Temperature Adjustment (for control panel yellow buttons) – DO NOT PRESS THE LEFT (SCROLL) KEY. TAP the Up or Down Arrow key; a digit will be highlighted. Holding down the Up or Down Arrow keys can change the highlighted digit to increase or decrease its setting. To change to another highlighted digit, TAP the Up or Down Arrow key. When the desired digit is highlighted, HOLD DOWN the Up or Down Arrow key to change the Control Point setting. After the proper Control Point is set, no further action is necessary. The display will return to the current temperature in about 10 seconds.

Control Point Temperature Adjustment (for control panel grey buttons) – PRESS and HOLD * key. The display will show the Set Point Temperature. The digits can be changed to the desired value by pressing the up and down arrows while holding the * key.

Control Point Temperature Adjustment (for control panel RKC SA200 buttonless) – Turn the HotBlock on and wait until the display shows the current block temperature (green digits). To change the set temperature, TAP the SET key once. The far right digit of the set value display (orange digits) will flash, indicating the flashing digit can be changed from 0 to 9 by using the up/down arrow keys. PRESS/TAP the up/down arrow keys to reach the desired value for that digit. Use the SHIFT (<R/S) key to move to the next digit and change it using the up/down keys as desired. Continue until you have reached the desired control point temperature. TAP the SET key TWICE to store the new set.

NOTE: Maximum temperature of polypropylene containers is 130°C.

Calculations:

Please consult SOP-IO-012 for calculation procedures.

Statistical Information/Method Performance:

Consult LOM-SOP-ES-203 and LOM-SOP-ES-207.

Quality Assurance/Quality Control:

Perform a method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample with every digestion batch (20 samples or less).

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Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035

Reference:

1. Method 5035, Revision 0, SW-846, U.S. EPA, December 1996.
2. EPA Method 5035A, Draft Revision 1, SW-846, U.S. EPA, July 2002.
3. Method AK101 – For the Determination of Gasoline Range Organics Version 4/8/02.
4. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
LOM-SOP-ES-225	Reagents and Standards
LOM-SOP-LAB-220	Laboratory Notebooks, Logbooks, and Documentation
SOP-SS-017	Preservation and Bottles Room Preservative Traceability

Scope:

This SOP will cover the preparation of vials to be used when preparing soil samples for low and high concentration volatile analysis. It will also cover preparing soil samples that have been collected in an appropriate sampling device for analysis, such as the Encore™sampler.

Basic Principles:

The sample is collected in a coring device (Encore™) and extruded into a pre-weighed volatile-free container and preserved within 48 hours of collection. The weight of the container, preservative, and soil is then captured and the net weight of the sample is calculated and captured in the Volatile Preparation program in Parallax.

Personnel Training and Qualifications:

The initial training consists of observing the procedure being carried out by an experienced analyst allowing for questions and feedback. Following the initial training, experienced analysts are available as a resource until no longer required. Analysts are considered proficient when the procedure can be carried out independently.

Interferences:

Sample contamination could occur if the sample preparation is not done in a volatile free environment; therefore, this process must be performed in one of the designated volatile free laboratories. Samples can also become contaminated by diffusion of volatiles through the sample vial septum. A trip blank carried through sampling, storage and handling can act as a check of such contamination.

Safety Precautions:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Methanol is flammable. Containers of this solvent must be kept away from any sources of open flames or sparks. Vials containing methanol must be stored in explosion-proof refrigerators.

Sodium bisulfate is an acidic solution. Normal protective equipment such as lab coat, gloves, and glasses should be used when working with these vials.

Sample Handling:

The Encore™ sampling device and the sample containers used to preserve the soil must be refrigerated at 2° to 4°C.

Apparatus and Equipment:

Alternate weights and volumes may be used as long as the final concentrations remain the same. See LOM-SOP-ES-225 for proper labeling documentation.

1. 40-mL vials with stir bars, Teflon™-lined low-bleed septa, and screw caps. SciSpec Catalog #376740-MB or equivalent
2. Encore™ sampler, or equivalent
3. Repipette capable of dispensing up to 25 ± 0.25 mL, or equivalent
4. Analytical balance capable of weighing 0.01 g
5. Preprinted labels with vial ID code
6. Extruder tool for EnCore™ sampler

Reagents and Standards:

The sodium bisulfate and methanol used must have been previously tested and approved for use by the labs. See SOP-SS-017 for further information.

1. Methanol – Purge and trap grade, store at room temperature and re-analyze yearly. The methanol used must have been previously tested and approved for use by the labs. See SOP-SS-017 for further information.

2. 8260A/B Surrogate Mix, Restek Catalog #30340 (2500 µg/mL) or equivalent. Store at –10 to –20°C. This standard may be used as is or may be diluted in methanol to a final concentration of 2.5 µg/mL. This standard is used for the GC/MS analyses.

3. Custom a,a,a, trifluorotoluene (TFT), Restek Catalog #54357 (15,000 µg/mL) or equivalent. This standard may be used as is or may be diluted in methanol to a final concentration of 750 µg/L. Store at –10 to –20°C for up to one month. This standard is used for the GC analyses.

4. Sodium hydrogen sulfate anhydrous powder, Fluka, Catalog #2316657 or equivalent. Store at room temperature and re-analyze yearly. If compounds are detected above the method detection limit (MDL), prepare another vial and repeat the analysis. If compounds are still detected above the MDL, a new container must be tested and used.

5. Sodium Bisulfate Solution – prepared by diluting 200 ± 5 g of the sodium hydrogen sulfate anhydrous into 1000 mL of deionized water in 1000-mL volumetric flask. Cap and invert at least 3 times to mix. Store at room temperature and re-analyze every six months if supply remains. If compounds are detected above the method detection limit (MDL), repeat the analysis. If compounds are still detected above the MDL, remake the solution and test before using.

6. Deionized Water – ASTM Type II (water from our in-house deionized system is acceptable)

Procedure:

NOTE: The Parallax VOA Prep application integrates a PC with an analytical balance to collect data directly from the balance. It organizes the data, performs calculations, and stores final results in the Laboratory Information Management System.

The VOA Prep application should be used whenever possible for this procedure to facilitate data transfers and other tracking. However, data may still be recorded traditionally in a logbook. Refer to LOM-SOP-LAB-220.

A. Preparing vials

1. Sodium bisulfate solution vials (low concentration Analysis #8389) used in preparation for Option A.

   a. Add 5 mL of sodium bisulfate solution to a clean 40-mL vial with a magnetic stir bar.

   b. Seal the vial with a screw cap and septum seal. (Septum seal must be able to withstand an acidic solution—low bleed septum recommended).

   c. Label the vial with a tracking number and capture the information using the Volatile Prep application.

2. Deionized water vials (used when effervescence occurs during preparation for Option A. Reference EPA Method 5035A, Draft Revision 1, SW-846, U.S. EPA, July 2002)

   a. Add 5 mL of deionized water to a clean 40-mL vial with a magnetic stir bar.

b. Seal the vial with a screw cap and septum seal. (Septum seal must be able to withstand an acidic solution—low bleed septum recommended).

c. Label the vial with a tracking number and capture the information using the Volatile Prep application.

3. Methanol vials (high concentration Analysis #8390, 6174, and 7578) used in preparation for Option B.

a. Add required amount of methanol with 8260 surrogate (pre-made solution) to a clean 40-mL vial.

b. Seal the vial with a screw cap and septum seal.

c. Label the vial with a tracking number and capture the information using the volatile prep application.

4. Methanol vials (high concentration Analysis #0065) used in preparation for GC/MS CLP analysis.

a. Add required amount of methanol to a clean 40-mL vial.

b. Seal the vial with a screw cap and septum seal.

c. Label the vial with a tracking number and capture the information using the volatile prep application.

5. Methanol vials (high concentration Analysis #6117 and 6130) used in preparation for GC analysis.

a. Add required amount of methanol with TFT surrogate (pre-made solution) to a clean 40-mL vial.

b. Seal the vial with a screw cap and septum seal.

c. Label the vial with a tracking number and capture the information using the volatile prep application.

B. Preparing the sample

Method 5035 calls for preservation of the soils in the Encore™ samplers within 48 hours of collection. Samples for low concentration analysis require three replicate collections. Two of these will be prepared into sodium bisulfate and one into methanol as a back up in case we need to change to high level. If the sample effervesces in sodium bisulfate, those two replicates will be prepared into deionized water and frozen. High concentration analysis only requires one collection which is then prepared into methanol. Samples are stored in a refrigerator/cooler at 2° to 4°C prior to preparation. To ensure that the samples remain cold, only take samples to the preparation area that can be processed in a short period of time.

NOTE: The methanol vial must be prepared first to ensure at least one vial is available for each required solution.

1. Sodium bisulfate solution vials

NOTE: Take 5 to 6 grams of the sample that was not collected in an EnCore™ sampler and place it in 5 mL sodium bisulfate solution. Check the pH of the sample in solution to ensure that it is <2. If it is not, add enough sodium bisulfate to bring the pH <2. Also check for effervescence. If a rapid or vigorous reaction occurs, prepare the EnCore™ collections into vials that contain a stir bar and 5 mL deionized water and then freeze at -10°C or colder. However, samples to be analyzed under the Ohio VAP regulations/requirement are **NOT** to be frozen under any circumstances.

At that point the client service representative should be contacted to check with the client and see how they would like us to proceed. There are two options at that point: 1) run the Methanol-preserved vial at high-level concentrations, 2) run the frozen in deionized water vial at low-level concentration.

a. Weigh a prepared vial to the nearest 0.01 g. Capture the weight.

b. Extrude the sample from the sampling device into the vial and immediately recap the vial.

c. Weigh the vial which now contains the soil to the nearest 0.01 g and capture the weight.

d. The weight of the soil is determined by a calculation done by the volatile prep program based on subtracting the weight of the prepped vial from the weight of the vial with soil. Note in the comment section if the Encore™ sampling device was not properly sealed or if the device was not full causing a low weight. The volatile prep application will generate an e-mail and send it to the appropriate client services representative.

e. Record any unusual observations about the sample.

2. Methanol vials

a. Weigh a prepared vial to the nearest 0.01 g. Capture this weight.

b. Extrude the sample from the sampling device into the vial and immediately recap the vial.

c. Weigh the vial, which now contains the soil to the nearest 0.01 g, and capture this weight.

d. The weight of the soil is determined by a calculation done by the volatile prep program based on subtracting the weight of the prepped vial from the weight of the vial with soil. Note in the comment section if the Encore™ sampling device was not properly sealed or if the device was not full causing a low weight. The volatile prep application will generate an e-mail and send it to the appropriate client service representative.

e. Record any unusual observations about the sample.

C. Deliver sample to the labs

Once the samples are prepared into either sodium bisulfate or methanol, they may be transported to the laboratory that will be analyzing them. Each department will have a designated-off spot that is refrigerated. A copy of the associated data must accompany the vials to the department.

Calculations:

Calculation of sample weight : Wn = Ws – Wf

Where:

Wf = weight of container + solution (first weight)

Ws = weight of container + solution + soil (second weight)

Wn = net weight of soil sample

Statistical Information/Method Performance:

Not applicable to this procedure.

Quality Assurance/Quality Control:

The number of sampling devices requested from client services should include the appropriate amount of extra bottles to serve as QC for the volatile analyses requested. Generally, the low-level analysis requires a total of seven Encore™ sampling devises. Four are prepared in sodium bisulfate, two for the unspiked, one for the matrix spike, and one for the matrix spike duplicate. Three are prepared in methanol, one for the unspiked, one for the matrix spike, and one for the matrix spike duplicate. Other numbers of sampling devises may be requested for QC purpose at the client's request.

Revision Log:

Ver. #	Effective Date	Change
01	12/23/97	New
02	01/22/98	Major changes are as follows: • Checking pH in sodium bisulfate • If sample effervesces, prepare into deionized water

Ver. #	Effective Date	Change
03	04/14/99	Major changes are as follows: • Changed the wording in the Scope section • Made some clarifications under preparing vials • Under section C, 1, added freezing samples at -10°C or colder • Added section on contacting the client representative to see how to proceed with samples that effervesce (section C, 1) • Added attachments on sampler requirements
04	10/21/99	Major changes are as follows: • Added the following sentence to Procedure C, Preparing the Sample: Method 5035 calls for preservation of the soils in the Encore samplers within 48 hours of collection.
05	04/12/00	Major changes are as follows: • Procedure C. – Removed "Not accepted by USEPA"
06	10/02/01	Major changes are as follows: • Cross Reference section added • Procedure – Added B.1.e. and B.2.e. • Incorporated Procedural Amendment #1
07	05/30/03	Major changes are as follows: • Rev. Log 06 – Change sections C.1.e and C.2.e. to section B.1.e. and B.2.e • Updated to Level 3
08	12/07/05	Major changes are as follows: • Updated Basic Principles, Interferences, Safety Precautions and Waste Handling, Sample Handling, Reagents and Standards, Procedure, and Quality Assurance/Quality Control sections • Incorporated Procedural Amendment #1 • Removed Attachment I

Ver. #	Effective Date	Change
09	FEB 2 8 2006	Major changes are as follows: • Updated Procedure section

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020806

Prepared by: _____ Date: 2/10/06
Specialist, Group Leader

Approved by: _____ Date: 2-10-06
Sample Support Management

Approved by: _____ Date: 2/14/06
Quality Assurance

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

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Analysis # 8389, 8390, 6130, 6117, 6174, 7578, 7320, 0065
Revision 09 PA #1
Procedure Effective Date: 02/28/06
PA Effective Date: FEB 2 8 2006

Procedural Amendment #1

Procedure Title:

Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035

Reasons for addition(s) or change(s): Correction

Samples or project affected: All

List change(s) or addition(s) (specify which section):

Procedure: *A.4. Replace the words GC/MS CLP Analysis with GC Wisc. GRO analysis.*

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022106

Prepared by: _____ Date: 2/21/06
Specialist, Group Leader

Approved by: _____ Date: 2-21-06
Sample Support Management

Approved by: _____ Date: 2/23/06
Quality Assurance

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Synthetic Precipitation Leaching Procedure (SPLP) Zero Headspace Leachates

Reference:

Method 1312, *Test Methods for Evaluating Solid Waste*, USEPA SW-846, September 1994.

Cross Reference:

Document	Document Title
SOP-TL-001	Glassware Cleaning for Leachate Extractions
SOP-TL-002	Leachate Blank Evaluations

Scope:

This method is used to determine the mobility of volatile organic contaminants in potentially hazardous waste. This extraction is performed over an 18-hour period.

Method Summary:

For liquid waste containing <0.5% solids, the SPLP extract resulting from the filtration of the waste through a 0.6- to 0.8-µm glass fiber filter in a zero headspace extractor (ZHE) is defined as the filtrate.

For waste containing >0.5% solids and some liquid, the waste is filtered through a glass fiber filter in the ZHE and the filtrate is collected and stored in a Tedlar® bag for later use. The solid left in the ZHE is then extracted with a volume of extraction fluid at 20× the weight of the solid. After extraction, the leachate is filtered through a glass fiber filter in the ZHE into the Tedlar® bag combining the final and initial filtrates.

For waste containing >0.5% solids which yield no liquid, 25g of the sample is extracted with a volume of extraction fluid at 20× the weight of the sample in the ZHE. The

sample is then extracted and filtered through the ZHE and the leachate is defined as the filtrate.

Glassware Cleaning:

See SOP-TL-001

Sample Handling:

Samples must be collected with no headspace and stored at 2° to 4°C. The holding time for sample extraction is 14 days from the time of collection.

Interferences:

Any interference that may be encountered during analysis is discussed in the individual analytical methods.

Personnel Training and Qualifications:

The technician using this method should be trained by a qualified technician, read and understand this SOP, and perform these procedures at least twice in that person's presence before they may be considered qualified. This training shall be documented in the employee's training manual.

Apparatus:

1. Tumbler – capable of rotating at 30 ± 2 rpm in an end-over-end manner
2. Zero headspace extractor (ZHE)
3. Glass fiber filter (0.6- to 0.8-µm pore size 90mm in diameter)
4. Tedlar® bags – 0.5 or 1.2L

5. Vacuum Filtration system (vacuum flask, filter holder (gooch), hose)
6. 48-lb. torque wrench (±4 lb.)
7. Poly extraction fluid transfer bag
8. Laboratory balance – capable of weighing to 0.01g
9. Graduated cylinders – Class A, assorted sizes
10. Gooch crucibles
11. Drill
12. Ratchet wrench
13. 40-mL vials
14. Aluminum weighing pans
15. Ruler
16. pH Meter – Orion Model 210A or equivalent – Capable of 0.01pH unit display
17. Septa for 40-mL vials – modified to allow a Teflon outlet tube to pass through

Reagents:

Extraction Fluid #3 – Deionized water

Safety Precautions:

Laboratory coats, gloves and safety glasses must be worn at all times when working with samples or reagents. Avoid skin contact or breathing the vapors from any reagents or samples. If samples are odorous or contain potentially hazardous material, use a ventilation hood. Discard, or send for repair, any glassware that is chipped or broken. When working on the ZHE use the ratchet wrench or cordless drill to prevent any wrist strain or injury. In case of injury notify your supervisor.

Preliminary Solids Determination:

If the waste appears to be liquid containing a very low percentage of solids, perform the solids determination. The Federal Register TCLP method defines percent solids as that fraction of waste from which no liquid may be forced out by an applied pressure.

Note: If the total solids determination was performed on the sample for the non-volatile leachate those values can be used.

1. Pre-weigh a filter in a gooch crucible and record the weight in the percent solids section of the volatile prefilter spreadsheet (See **Figure 1**).

2. Weigh out 25 ±0.1 g of the sample into a graduated cylinder and record the sample number and the weight of the sample plus the graduated cylinder on the Volatile_Pre-filter.xls spreadsheet, which is accessible on the DP28 computer via the desktop icon (shortcut).

3. Pour the sample into the vacuum filter apparatus, and slowly apply vacuum until no liquid flows through the filter.

4. Reweigh the graduated cylinder. Record the weight of the graduated cylinder and residue adhered to the cylinder. The spreadsheet will calculate the weight of sample filtered.

5. The material in the filter holder is defined as the solid phase (sludge cake and filter) of the waste and the liquid phase is the filtrate.

6. Weight the sludge and filter and record the weight on the spreadsheet.

7. The spreadsheet will determine the percent solids using the calculation listed below:

$$\%\ solids = \frac{Weight\ of\ sample\ recovered}{Weight\ of\ sample\ filtered} \times 100$$

8. If the percent solids is <0.5% print the spreadsheet and go to Procedure A.

9. If the percent solids is between 0.5% and 2.5%, dry the filter and solid at 100° -120° C until two successive weights yield the same value within ±1%. Record each weight on the spreadsheet. The percent dry solids are determined using the following calculation:

$$\%\ Dry\ Solids = \frac{Weight\ of\ dried\ solid\ recovered}{Weight\ of\ sample\ filtered} \times 100$$

10. If the percent dry solids is <0.5% print the spreadsheet and go to Procedure A. If the percent dry solids is >0.5% go to Procedure B.

Procedure:

A. Samples that contain <0.5% solids:

1. Assemble the ZHE and add enough sample to fill three 40-mL vials after filtration.

2. Filter the sample through glass fiber filters (0.6- to 0.8-µm) and collect the sample in the vials with **NO** headspace, unless the sample is from California. In that case, collect the final filtrate into a single labeled Tedlar® bag and deliver to the appropriate department. This filtrate is defined as the SPLP volatile extract.

3. Change the analysis #8792 to #1339 and add the appropriate comment in Parallax.

B. Samples that contain >0.5% solids and have a standing liquid phase:

1. Assemble the ZHE extractor.

2. Pre-weigh an empty Tedlar® bag and record the weight on the volatile prefilter spreadsheet (see **Figure 1**).

3. Weigh out a minimum of 25g of sample into an aluminum weighing pan and record the weight on the spreadsheet.

4. Pour the sample into the ZHE extractor. Re-weigh the aluminum pan used to weigh out the sample and record the weight of the residue and cup. The spreadsheet will calculate the weight of sample.

5. Attach the Tedlar® bag to the ZHE extractor and filter the sample through the ZHE extractor into the Tedlar® bag until the liquid stops flowing. Reweigh the Tedlar® bag with the liquid and record the weight. The spreadsheet will calculate the weight of the liquid in the bag and the weight of the solid in the extractor.

Note: If the filter in the ZHE gets clogged DO NOT change it. Any sample left in the ZHE extractor is considered to be the solid. If the liquid is not water-soluble, a larger amount of sample will have to be filtered, because both layers will have to be analyzed separately. You will need to make sure you have enough solids to complete the SPLP analysis on the sample.

6. Record the weight of the solids under weight of sample extracted in the data log.

7. The necessary volume of extraction fluid (20× the weight of the sample to be extracted) should be measured into the fluid holding bag using a graduated cylinder. Connect the fluid holding bag to the ZHE and open the valves on both the transfer line and the ZHE. Slowly add Extraction Fluid #3 (extraction fluid #3 is always deionized water for volatile analysis) by turning the ZHE piston downward with a drill.

8. Once the entire volume of extraction fluid has been transferred from the bag to the ZHE, close the valve on the ZHE and hand crank the piston with the 48-lb. torque wrench until it clicks. Slowly open the valve to release any air that was trapped in the extractor. Close the valve when the liquid appears. Again crank the piston with the 48-lb. torque wrench until it clicks. Place the extractor on the tumbler and rotate at 30 ± 2 rpm, for 18 ± 2 hours. Record the tumbler ID in the data log. Ambient temperature must be maintained at 23° ± 2°C (69.8° to 77°F) during the extraction.

9. When the extraction is complete, connect the Tedlar® bag containing the initial filtrate to the ZHE unit and slowly open the valve on the bag. Quickly open and close the valve of the extractor while listening for an escape of gas. If no gas escapes, the extractor had a leak during the extraction and the sample will need to be repeated. In addition, the extractor must be checked for leaks by pressurizing it to 50 psi and submerging it in water. Check for air bubbles coming from any of the fittings. If air bubbles are present, check all fittings and inspect O-rings and replace them if necessary. Reassemble and

pressurize the extractor and repeat the submersion leak check. If the extractor continues to leak, remove it from service and contact the manufacturer.

10. Filter the entire sample extract through the filter already in the ZHE into the Tedlar® bag unless the filtrate is not water-soluble. If the initial filtrate is not water-soluble, **DO NOT** try and mix them together. They will have to be analyzed separately. Notify your supervisor so that they may contact the appropriate person(s). Collect the final filtrate into three 40-mL glass vials with **NO** headspace, unless the sample is from California. In that case, collect the final filtrate into a single labeled Tedlar® bag and deliver it to the appropriate department.

C. Samples that contain >0.5% solid and no liquid:

1. Assemble the ZHE extractor.

2. Weigh out 25±0.1 g of sample into an aluminum weighing pan and record the weight in the data log under sample weight.

3. Pour the sample into the ZHE extractor.

4. Fill the fluid holding bag with extraction fluid 3.

5. Add 500 mL of extraction fluid to the extractor by connecting the transfer line from the bag to the extractor. Open the valves on the extractor and the line. Slowly turn down the piston of the extractor using a drill.

   Note: If less than 25.0g of sample is used for the extraction, the exact volume of extraction fluid needed (20× the weight of sample) for the sample, must be measured into the fluid holding bag. See Procedure B.7.

6. When the piston reaches the bottom of the extractor, close the valves on the extractor and the bag. Detach the transfer line and slowly crank the piston upwards using a 48-lb torque wrench until it clicks. Slowly open the valve and release any air trapped in the extractor. Close the valve when the liquid appears. Again, crank the piston with the 48-lb torque wrench until it clicks.

7. Verify that 500 mL of fluid was added to the extractor by using a ruler to measure the length of the rod protruding from the bottom of the extractor. At least 4 3/8" of the rod must be visible or the extractor does not contain 500 mL of fluid. If the required length is not visible, add additional fluid following Steps C.4. – C.5. until the extractor contains 500 mL of extraction fluid.

   Note: This step should only be performed when 25.0g of sample was weighed in step C.1.

8. Place the extractor on the tumbler and rotate at 30 ±2 rpm for 18 ±2 hours. Record the tumbler ID in the data log. Ambient air temperature must be maintained at 23°C (69.9° to 77°F) during the extraction.

9. When the extraction is complete, attach a Teflon® tube to the outlet valve of the extractor. If the sample is not from California, place the tube through a modified septum and into a 40-ml vial. If the sample is from California, attach the tube to a Tedlar® bag. Quickly open and close the valve of the extractor while listening for an escape of gas. If no gas escapes, the extractor lost pressure during the extraction and the sample will need to be repeated. If the extractor has leaked, it must be checked for leaks while empty by pressurizing it to 50 psi and submerging it in water. Check for air bubbles coming from any of the fittings. If air bubbles are present, check all fittings and inspect O-rings and replace them if necessary. Reassemble and pressurize the extractor and repeat the submersion leak check. If the extractor continues to leak, remove it from service and correct the source of the leak.

10. Using a rachet, turn the piston and filter the sample through the filter already in place in the ZHE. If you suspect the internal filters have ruptured, place an on-line filter on the outlet tube from the ZHE. Filter the sample into at least three 40-mL glass vials with **NO** headspace, unless the sample is from California. In that case, collect the final filtrate into a labeled Tedlar® bag and deliver it to the appropriate department.

Quality Assurance:

1. A minimum of one blank must be performed for every 20 samples extracted in a day. This blank is not the same as the vessel blank.

2. A blank must be performed after every 20× an individual ZHE is used. A logbook of vessel usage is kept. Each time a vessel is used, record the date and sample number. After 20 uses, prepare a blank in that vessel and record the blank number in the logbook. Every blank will be evaluated for contamination using the guidelines in SOP-TL-002.

3. Record all sample numbers and the batch blank number in the batch logbook.

4. All instruments used in this procedure should be calibrated according to an approved laboratory plan.

5. All quality control measures described in the appropriate analytical methods shall be followed.

Revision Log:

Ver. #	Effective Date	Change
01	08/06/98	New
02	08/27/01	Major changes are as follows: • Cross Reference section added • Apparatus – Clarified • Glassware Cleaning – Section added • Procedure/Preliminary Solids Determination – Added spreadsheet information, clarified addition of extraction fluid • Quality Assurance – Clarified, incorporated Procedural Amendment #1 • Added Figure 1
03	NOV 1 0 2004	Major changes are as follows: • Updated Format • Updated filtration procedure • Updated Figure 1

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100504

Prepared by: _____ (1092) Date: 10-6-04
Senior Chemist Coordinator

Approved by: _____ Date: 10.25.04
Organic Extraction Management

Approved by: _____ Date: 10/27/04
Quality Assurance

Figure 1

Sample # 1234567
Date:
Technician:

Percent Solids Determination

Liquid/Solid Separation	
Weight of filter paper	1.00
Weight of sample and grad	1.00
Weight of grad and residue	1.00
Weight of sample filtered	0.00
Weight of solid plus filter	1.00
Weight of solid recovered (g)	0.00
Percent Solids (Wet)	#DIV/0!
Weight of dried sample and filter	1.00
Weight of dried sample and filter	1.00
Weight of dried sample and filter	1.00
Weight of dried sample	0.00
Percent Solids (Dry)	#DIV/0!

Volatile Leachate Prefilter

Liquid/Solid Separation	
Weight of Tedlar bag (g)	1.00
Weight of sample and cup (g)	1.00
Weight of residue and cup (g)	1.00
Weight of sample (total) (g)	0.00
Weight of bag and liquid (g)	0.00
Weight of liquid in the bag (g)	-1.00
Weight of sample in vessel (g)	1.00
Volume of ext. fluid to add (mL)	20

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MC-IO-014.06
Supersedes Date: 08/17/04
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 AUG 3 1 2006

Vapor Generation for Cold Vapor Mercury Method Using the Leeman Labs PS200

Approvals:

Prepared by: Jennifer L Moyer Date: 08/14/06
Chemist/Coordinator

Approved by: Date: 8.15.06
Metals Management

Approved by: Elaine Stoltzfus Date: 8/17/06
Quality Assurance

Revision Log:

Ver. #	Effective Date	Change
00	10/05/95	Previous issue
01	01/03/00	Major changes are as follows: • Updated method reference. • Deleted Quality Control section. • Added Quality Assurance section referencing SOP-IO-005. • Added Personnel Training and Qualifications section.
02	08/04/00	Major changes are as follows: • Cross Reference section added • Figure 1 – ICV is listed as 2.00 , number should be 2.50
03	03/17/03	Major changes are as follows: • Updated Reference section to include CLP 5.2
04	03/20/03	Major changes are as follows: • Added section b. to Procedure
05	08/17/04	Major changes are as follows: • Deleted autosampler setup note • Updated figure 1
06	AUG 3 1 2006	Major changes are as follows: • Updated format to comply with LOM-SOP-LAB-201.05. Moved the Approval and Revision Log sections to the front of the document.

MCIO014_06.DOC
080906

Reference:

The method described in this maintenance and calibration is adapted from the following references.

1. Method 7470A (waters) and 7471A (solids), *Test Methods for Evaluating Solid Waste*, USEPA SW-846, modified, September 1994.

2. USEPA CLP SOW ILM04.0, Exhibit D, CLP-M, D4.

3. USEPA CLP SOW ILM05.2, Exhibit D/Mercury.

4. Method 245.1, *Methods for Analysis of Water and Wastes*, USEPA 600/4-79-020, Rev. March 1983.

5. Method 245.1, *Methods for the Determination of Metals in Environmental Samples*, Supplement I, EPA-600/R-94/111, May 1994.

6. Leeman Labs suggested operation and maintenance.

Cross Reference:

Document	Document Title
SOP-IO-005	Quality Control Procedures for Mercury
SOP-IO-007	Preparation of Standards and Solutions
SOP-IO-011	Inorganic Analysis Safety and Waste Handling Procedures

Purpose:

The purpose of this procedure is to outline the proper techniques for the operation, calibration, and maintenance of the Leeman Labs, PS200 Automated Mercury Analyzer.

Scope:

The Leeman Labs PS200 Automated Mercury Analyzer utilizes continuous flow technology with drying of the sample vapor for the analysis of mercury by automated vapor generation. The dry vapor enters one path of a heated double path optical cell, which has been optimized for fast response (small diameter), and sensitivity (long length). Mercury is measured using a solid state detector with a wide dynamic range and a mercury source that delivers a stable source of emission at 254 nm. The signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions. This procedure offers advantages in sensitivity and precision over conventional CV-AAS systems by a factor of approximately 10 times.

This procedure is applicable to the determination of mercury in waters, wastewaters, leachates, and soils by vapor generation.

Personnel Training and Qualifications:

All personnel that are responsible for operating the Leeman Labs PS200 will have a working knowledge of both operating and maintenance procedures for the instrument. Training to achieve this knowledge will consist of reading related departmental SOPs and instrument operations manuals as well as hands-on experience. A trainee will first observe a trained analyst perform the functions, then the trainer will supervise and review the trainee's performance and data. Final qualifications will require acceptable performance on quad studies for this or an equivalent procedure.

Safety Precautions and Waste Handling:

Refer to SOP-IO-011.

Standard and Reagent Preparation:

Refer to SOP-IO-007.

Procedure:

A. Instrument setup

1. Leeman Labs Automated Mercury Analyzer

The Leeman Labs PS software has been set up using "hotkeys" to help with moving around in the software and macro utilization. (Macros are small programs used to operate the instrument.) The hotkeys are the capitalized letters that are highlighted in red on the computer screen.

a. System conditioning

If the instrument and computer are off, turn ON the power to the instrument (green button). Also turn ON the power to the computer and printer. Once the computer has initialized and a C:\ICP\ prompt is obtained, type "PS" and press ENTER. This will initialize the PS instrument software. The system is now ready for the initial setup. If the instrument was shut down completely, the optical cell must be reconnected. To reconnect the optical cell, remove the front cover from the instrument by sliding the cover up and lightly pulling it out. Remove the two screws from the retaining brackets and carefully slide the cell out of its holder. Connect the gas lines on the left side of the cell to their respective connections. Check that all connections are secure for the gas, lamp, detector, and cell heater. Place the cell in its holder and tighten the retaining brackets to hold the cell in place. Replace the front cover to the instrument.

Turn ON the power to the mercury lamp (blue button). The lamp will take approximately 20 minutes to stabilize. Press F10 to stop and exit any previously running macro. Press F1 to go to the Main menu. Use hotkeys by typing "U" to select Utilities and then by typing "G" to select diaGnostics. Using the down arrow, move the cursor to Tip Home and press ENTER. This will move the autosampler tip to the rinse position and raise the tip out of the rinse tank. Remove the sample trays and the rinse tank from the autosampler. Rinse the rinse tank with deionized water several times and refill the rinse tank with a 1% nitric acid solution (1% HNO_3).

Change the drying tube (located below the liquid gas separator) with a fresh, loosely packed tube from the prep room desiccator by unscrewing the gray lock nuts, removing the old tube, and replacing it with the fresh tube. Tighten the lock nuts and pull lightly on them to ensure a snug fit. If a seal is not observed, remove the O-rings from the lock nuts and rinse them with deionized water. Replace the drying tube in its holder on the instrument.

NOTE: It is very important that the tube is packed loosely so that the carrier gas can flow freely through the quartz wool and magnesium perchlorate.

Replace the rinse tank and sample racks on the autosampler. Press F1 for Menu, then type "I" for Instrument, "O" for Operations, and then "T" for Tip to Rinse. This will lower the autosampler tip into the rinse tank. Check the pump tubing for excessive wear by flipping the locking hub down and lifting up on the pump cassette. Visually check the tubing for excessive wear. Under normal daily operation, the tubing should last approximately 1 week. To replace the tubing, disconnect old tubing and discard and obtain a new set of tubing from the parts drawer. All tubing should be replaced together. The pump tubing is color coded for easy replacement. The sample tubing is black with a blue tab, the reductant tubing is clear with a red, orange, or yellow tab (0.16 cc/m), and the

2 waste tubes are clear with a gray tab (1.00 cc/m). The tubes are placed in separate cassettes with the sample tube closest to the instrument, followed by the reductant tube and finally the 2 waste tubes. To secure the tubing to the pump head, slide the tubing through the plastic clips at the bottom of the cassette until the tab is secure. Hold the tube taut (not stretched) and slide the loaded cassette onto the pump head.

Lock the clamp by pulling the locking hub up. Repeat for the remaining tubes and connect the ends to the respective connections. For new tubing the "coldstrt" macro should be used.

Once the above initial system checks are complete there are 2 system conditioning procedures for the instrument. If the instrument has been shut down completely, usually over a long weekend or if new tubing is used, press F2 for Macro, type "coldstrt," and press ENTER. The system will run approximately 2 hours, conditioning the pump tubing, lamp, and cell. When the macro is finished it will beep and display "Operation Complete" in red flashing letters at the bottom of the computer screen. The system is now ready to be optimized for automated analysis. If the instrument is in the overnight mode, used when daily operation is necessary or when tubing has previously been conditioned, press F2 for Macro, type "warmstrt," and press ENTER. The system will run approximately 15 minutes, conditioning the pump tubing, lamp, and cell. When finished, it will beep and display "System Ready" in red flashing letters at the bottom of the computer screen. The system is now ready to be optimized for automated analysis.

b. Optimization

Once the system is conditioned the cell must be optimized. Remove the front cover of the instrument and the large hex wrench from the lid of the cover. In the computer software, press F1 to go to the Main menu. Select Utilities and then diaGnostics by typing "U" and then "G." Using the down arrow, move the cursor to Aper Test and press ENTER. A correctly optimized system value is 0. The value displayed on the screen should be ±100. If adjustments are required, turn the lower hex screw clockwise Λ-turn for a positive adjustment and counterclockwise Λ-turn if a negative adjustment is required. After the adjustment is done, press ENTER to recheck the value. Adjust the aperture until the value is between ±100. Replace the hex wrench and instrument cover.

c. Run setup

Once the cell has been optimized, the instrument is ready for sample analysis. Press F1 for Menu, type "P" for Protocol, and "G" for Get. It will ask you for a protocol name. The protocols have been set up for those operating the instrument as initials-employee number (e.g., JKG-201). A default protocol has been set up as TEST. Type in the protocol name and press ENTER. It will prompt you for a folder name. The folder name is the file where the run information will be stored. Type in the folder name as follows and press ENTER: year, day, run number (e.g., 9403201 for the first run of day 032 of 1994). Press F1 to return to the Main menu.

d. Autosampler setup

To set up the autosampler, type "A" for Autosampler and "R" for Rack Entry. It will prompt you for a rack name. There are 2 sample racks called stations, which hold 44 samples each. The standards and check standards are located on their own rack. If less than 44 samples are to be used, only one station is required to be defined. Enter a distinctive rack name such as the day number and 01 or 02 and press ENTER. The INSERT key is used to move between the entry modes. The 3 entry modes are single cells down ("cell entry"), single cell across ("row entry"), and full column ("column entry"). The current entry mode is displayed at the bottom right corner of the computer screen. In cell entry mode, move the cursor to the cup ID column using the arrow key. Enter the labels, up to 10 characters, and press ENTER. Enter PBW or PBS for blanks, LCSW or LCSS for lab controls, and sample numbers followed by a space then U, D, R, or M for background, duplicate, spike, or matrix spike duplicate, respectively. Once the sample labels have been entered, the extended ID must be entered. The extended ID includes the initial weights or volumes and batch numbers. In the column entry mode, type "R" for Range and enter the range of samples for the first batch. Type "E" for Extended ID (the full range that was selected will be highlighted). Enter the most common initial weight or volume followed by a space and then the full batch number and press ENTER (e.g., 8 mL 940250821001). To edit values in either sample or extended ID columns, select either cell or row entry mode and move the cursor to the cell to be edited. Delete the value to be changed, retype the corrected value and press ENTER. Once the entries for all samples and batches are complete, return to the Main menu by pressing F1. The defined sample rack must be loaded into the autosampler setup to be run. Type "A" for Autosampler, "S" for Setup, and 1 for Station Rack Number 1 or 2 for Station Rack Number 2. You will be prompted for a rack name, enter the rack name, which corresponds, to the sample IDs you will be running. Enter the beginning cup position and the end cup position. Return to the Main menu by pressing F1. Load the standards,

check standards, and samples into the appropriate locations (see Figure 1).

2. Analysis of samples

   a. The instrument is now ready for automated analysis. To start the run, press F2 for Macro. You will be prompted at the top of the computer screen for a macro name. Type "CLPHG" and press ENTER. The instrument will go through several checks and setups before starting a calibration. The CLPHG macro has been developed to check the correlation coefficient of the curve, run appropriate check standards at proper intervals, and check the percent recoveries of the calibration check samples. If, for any reason, the checks fall outside the windows required, a recalibration and reread of any associated samples and check samples in the bad blocks will automatically be performed. Recalibration due to failure will repeat three times. If any of the checks fail after the third recalibration, the instrument will stop and an error message will be displayed.

   b. Stannous chloride is added to the samples via a "Y" connection in the pump tubing. The peristaltic pump then carries the sample/stannous mix to the reaction cell. Argon gas is used to sweep the volatile mercury into the absorption cell in the optical path of the atomic absorption spectrophotometer. The mercury (Hg^{++}) is reduced with stannous chloride (Sn^{++}) to liberate mercury metal and Sn^{+4}.

      Detailed instructions for the complete instrument setup are found in the *Leeman Labs PS200 Automated Mercury Analyzer Manual*, beginning with Section 3.0.

3. Instrument shutdown and cleanup

   a. Overnight shutdown

(1) Press F10 to stop and exit any running macro.

(2) Remove cap from the reductant bottle and place on rim of the rinse tank. Cap the reductant bottle with spare lid.

(3) Press F2 for Macro, type "overnight," and press ENTER.

(4) Turn OFF the power to the lamp.

(5) Remove all standard and sample tubes and place them on the washroom cart to be cleaned.

(6) Clean up any spills, which may have occurred during sample pouring or analysis.

b. Long-term shutdown (more than 3 days of no operation)

(1) Press F10 to stop and exit any running macro.

(2) Remove the rinse tank by following the steps for system conditioning. Rinse the tank with deionized water several times and fill it with deionized water. Replace the rinse tank on the autosampler.

(3) Remove cap from the reductant bottle and place it on rim of the rinse tank. Cap the reductant bottle with spare lid.

(4) Turn OFF the power to the lamp.

(5) Press F2 for Macro, type "shutdown," and press ENTER. The system will flush its lines with deionized water for several minutes.

(6) When finished, you will hear a beep and the message "Disconnect optical cell" will appear at the bottom of the computer screen. Disconnect the optical cell by removing the front cover of the instrument. Loosen the screws on the retaining brackets of the optical cell and gently slide the optical cell out of its holder. Disconnect the two gas lines on the left side of the cell and leave them hanging. Replace the optical cell in its holder and lightly tighten the retaining brackets. Replace the front cover of the instrument.

(7) Shut OFF the power to the computer, printer, and the PS200 instrument.

(8) Remove all standard and sample tubes and place them on the washroom cart to be cleaned.

(9) Clean up any spills, which may have occurred during sample pouring or analysis.

4. Maintenance

a. To clean and rejuvenate the drying tube, remove the quartz wool from the ends of the used drying tube and empty the magnesium perchlorate into a beaker. Dissolve the magnesium perchlorate in water and discard this solution as liquid waste in the acid waste carboy located in the washroom. Clean the drying tube by rinsing it with tap water and placing it in a sample rack to dry. Once the drying tube is dry, use a small piece of quartz wool to plug one end of the drying tube. Fill the tube with coarse magnesium perchlorate and plug the other end with the quartz wool. Place the freshly packed drying tube in the desiccator in the prep room.

b. The pump tubing should be replaced weekly under normal daily usage.

c. Lightly spray the pump head weekly with a lubricant.

d. Lubricate the autosampler weekly by placing several drops of a light oil onto a towelette and wiping down the rails.

e. On a monthly basis, check the optical cell and windows, and if needed, clean the optical cell with a soapy solution (one drop of liquid Ivory to 500 mL deionized water) and warm tap water. Rinse with deionized water and dry. To speed the drying of the optical cell, connect the heater plug to the optical cell with the windows off for several minutes. Clean the quartz windows with methanol and a piece of lens paper.

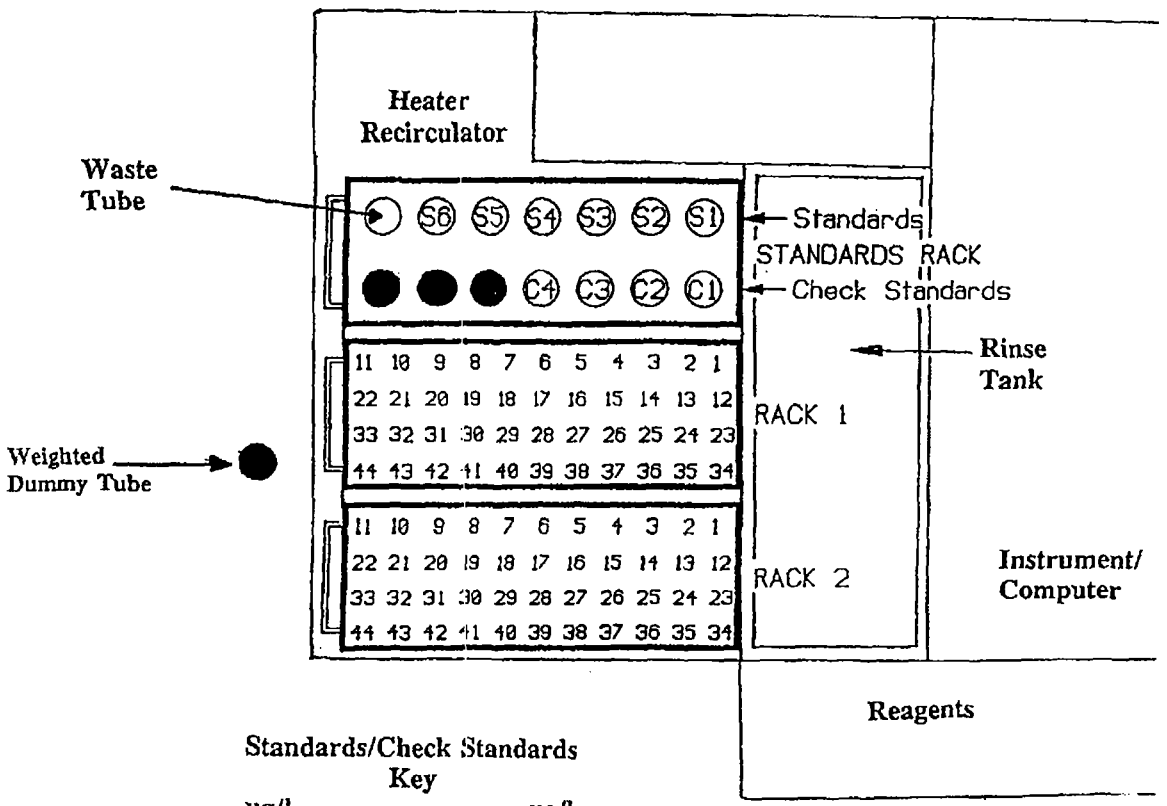
f. Document any maintenance in the Mercury maintenance logbook located next to the instrument.

NOTE: Detailed instructions for the maintenance and troubleshooting of the Leeman Labs Mercury Analyzer can be found in the *PS200 Leeman Labs Mercury Analyzer Manual.*

Quality Assurance/Quality Control:

Consult SOP-IO-005, for specific QC protocol and procedures.

Figure 1



Standards/Check Standards Key

	ug/l			ug/l
S1	0.00	C1	CCB	(0.00)
S2	0.20	C2	ICV	(2.50)
S3	0.50	C3	CCV	(1.00)
S4	1.00	C4	CRA	(0.20)
S5	2.50			
S6	5.00			

Maintenance for the TJA ICAP™ 61E Trace Analyzer Spectrometer

Reference:

ICAP™ 61E Trace Analyzer Operator's Manual, July 1993.

Cross References:

Document	Document Title
MC-IO-018	Operation of the Thermo Jarrell Ash ICAP 61E Trace Analyzer Spectrometer

Purpose:

The purpose of this procedure is to outline the proper maintenance of the Thermo Jarrell Ash ICAP™ 61E Trace Analyzer.

Scope:

This procedure will describe the steps involved in the maintenance of the Thermo Jarrell Ash ICAP™ 61E Trace Analyzer.

Documentation:

Any adjustment to an instrument, replacement of parts, etc., must be documented in the appropriate instrument logbook.

Personnel Training and Qualifications:

1. Review and understanding of this procedure
2. Trainee observing trained analyst performing the procedure
3. Trainer observing trainee performing the procedure

4. Documentation of critical steps in the training process

5. Demonstration of proficiency by being able to independently maintenance the ICP

Procedure:

I. Routine Maintenance:

A. Remove and clean the sample introduction system when instrument performance declines (See **Figure 1**).

1. Remove spray chamber and nebulizer.

a. Disconnect spray chamber from torch assembly by gently pulling out.

b. Disconnect nebulizer from spray chamber. (Gently, all nebulizers are very fragile.) We use both the Meinhard and Burgener Peek Mira Mist nebulizer. Do not touch or wipe the tip of the Burgener Peek Mira Mist nebulizer.

c. Disconnect argon supply and sample/internal standard/buffer tubing from nebulizer.

d. Clean spray chamber if residue is observed coating the sides. If cleaning is necessary, remove drain tube from spray chamber.

2. Remove and disassemble the torch.

a. Disconnect the coolant and auxiliary argon lines from the torch assembly. (This is done by pressing up on the red releases while pulling down on the tubing.)

b. Disconnect the torch assembly by loosening the black spring-loaded thumb screws at the top and bottom of the torch assembly. (Hold onto the torch assembly so it does not drop out.)

c. Pull the torch assembly out of the instrument.

d. Loosen and remove the two knurled metal nuts that hold the torch assembly together and disassemble the torch.

e. Remove the rubber O-ring that holds the injector tip in place and remove the injector tip by pushing it up through the torch base.

3. Prepare ultrasonic bath.

a. Empty any water which may be in the bath if water is dirty.

b. Make sure bath is at least ½ full with deionized water.

4. Clean torch, injector tip, and nebulizer.

a. Invert the torch in a 250-mL vacuum flask of 50% HCl and place in the sonicator for 10 minutes. (Position it in a manner that keeps the inner and outer tubes in the acid solution and the base is kept dry.) Rinse out torch tube with deionized water. (Be careful not to get a lot of water down into the base of the torch.) Carefully dry torch with a paper towel.

b. Place the injector tip in 50% HCl for 10 minutes. Rinse the injector tip with deionized water and carefully dry with a paper towel.

c. Place the nebulizer in 50% HCl for 10 minutes. (Do not place nebulizer in the sonicator.) If there is a visible clog, 0.13 diameter fishing line may be carefully inserted through the tip of the nebulizer to assist in removing the clog. Force the 50% HCl solution through the argon and sample inlets in the nebulizer and rinse with deionized water when

finished. (Make sure all of the water is out of the argon cavity of nebulizer.)

d. If spray chamber needs to be cleaned, place it in sonicator for approximately 5 minutes, then rinse it out with deionized water.

B. Reassemble the sample introduction system (see **Figure 1**).

1. Reassemble the torch.

a. Place the injector tip back in torch and fasten it with the black O-ring.

b. Reassemble metal torch mount, torch base, and PTFE support block with the two knurled metal nuts.

c. Remount torch into the instrument by inserting straight through the torch hole and the coils. Refasten the black spring-loaded thumb screws to the torch box.

d. Reconnect the auxiliary and coolant argon lines to the torch.

2. Reassemble the spray chamber and nebulizer.

a. Reattach drain tube to spray chamber making sure the metal clamp is around drain tube and glass outlet.

b. Fit the spray chamber back into the torch assembly. Slide the spray chamber into the torch assembly as far as it will go.

c. Reconnect argon supply and sample/internal standard/buffer tubing to the nebulizer.

d. Reinsert nebulizer into the spray chamber. Nebulizer should be mounted so that the nebulizer tip is flush with the inside wall of the spray chamber.

3. Ignite the plasma.

   See MC-IO-018.

4. Profiling through the ICP Manager software.

   a. Click one time on "INSTRUMENT" on the menu at the top of the screen. Highlight "AUTOMATED PROFILE" on the drop down menu, and click.

   b. Under "REPORTING NAME", click on the box next to As 189.042/2, to check it. Make sure that the vernier position listed on the right hand side of the window matches what the instrument dial is set to.

   c. Click on "NEXT"

   d. Click on "PROFILE"

   e. When the profile is complete, the peak position will be at the bottom of the window. If it is good (>-0.1 and <0.1), record the peak position, click on the print icon, click on "OK". Record the signal/background ratio number off of the printout as well as the peak intensity. Estimate the baseline and record. Click on "CANCEL", or "BACK" to take another profile at the same vernier position. If it is out (>0.1 or <-0.1), change Vernier position to the new one listed next to the peak position. Click on the save icon to keep Vernier position. Start over from the beginning. (The Vernier position is set by using the Hg Profile dial on the front instrument panel.)

C. Change pump tubing on peristaltic pump when the tubing shows wear. Inspect all tubing to insure that it is secure and in good condition. Periodically do visual check of the oil level in the vacuum pump and water level in the coolflow.

D. Clean the optics in the argon purged optical path periodically as needed.

1. Remove the argon purged optical path tube and nozzle.

a. Turn off the plasma and bring the instrument down.

b. Open panel door behind the plasma window on the front of the instrument and turn the crank (inside the door on the upper left hand side) clockwise until closed.

c. As a safety precaution, also turn off the HIGH VOLTAGE switch on the back of the instrument.

d. Remove the argon purged optical path tube screws. Hand loosen the horizontal plasma image adjustment control without moving the circular ring on the screw. (The circular rings are set to the previous alignment.)

e. Pull unit apart and twist off the nozzle.

2. Remove lenses and clean.

a. CAREFULLY pull out copper ring holding the lenses in place.

b. Remove lenses observing the fit of the two lenses. Flatter side of small lens fits against concave side of larger lens. The flat side of the larger lens goes in first, closest to the tip of the nozzle. See **Figure 2.**

c. Use lens paper with deionized water and mild soap to clean. Dry thoroughly with lens paper.

3. Reassemble the argon purged optical path tube and nozzle.

    a. Replace lenses one at a time and CAREFULLY squeeze in the copper ring to hold the lenses firmly in place. Be sure the lenses are replaced in the correct position (see **Figure 2**).

    b. Replace unit into instrument. Move torch exhaust stack on the top of the instrument enough to peer down into the exhaust outlet and line up the nozzle ¼" to ½" away from the torch.

    c. Turn high voltage switch (on the back of the instrument) back ON. Turn crank (inside the door on the upper left-hand side) counterclockwise until fully open.

4. Manual profile

    a. Bring the plasma up.

    b. In the ICP Manager Software, go into instrument/analog profile and select As to profile.

    c. Aspirating deionized water, turn screws supporting cone to find the lowest point on the meter (or the middle of the plasma). Set lock washers to hold new position.

II. Monthly Preventive Maintenance:

The following is to be done by the Maintenance Department every month (give or take a week):

1. Vacuum instrument air filters

2. Vacuum air intakes

III. Quarterly Preventive Maintenance:

Change vacuum pump oil and examine vacuum pump for possible problems.

IV. Semiannual Preventive Maintenance:

Examine, clean, and lubricate the moving parts on the autosampler.

Nonroutine Maintenance:

Examine the *ICAP™61E Trace Analyzer Operator's Manual*, Section 11, for more information on maintenance and equipment failures. Section 11 lists recommendations on cleaning the nebulizer if the steps in routine maintenance fail to solve the problem. Consult TJA service representative or applications chemist for further information on troubleshooting.

Revision Log:

Ver. #	Effective Date	Change
00	05/14/96	Previous issue
01	02/24/99	Major changes are as follows: • Added Personnel Training and Qualifications section • Added a section D under Routine Maintenance: Clean the optics in the argon purged optical path periodically as needed.
02	09/20/00	Major changes are as follows: • Cross Reference section added • B.3. Changed SOP reference to MC-IO-018
03	OCT 1 3 2004	Major changes are as follows: • Section B; Profiling instructions for the ICP Manager Software were added, Thermo SPEC software was deleted. • Clarifications throughout the SOP. • Updated to level 3 format

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Prepared by: Nina C Haller Date: 9-29-04
Chemist Coordinator

Approved by: [signature] Date: 9.29.04
Metals Management

Approved by: Dorothy M Love Date: 9/29/04
Quality Assurance

Figure 1

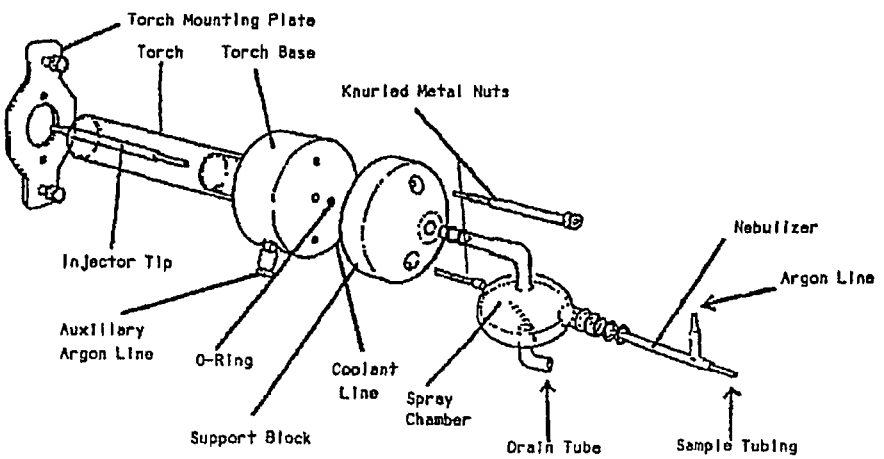
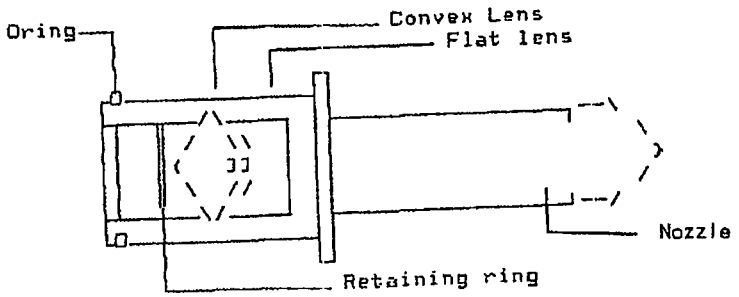

Torch Mounting Plate
Torch
Torch Base
Knurled Metal Nuts
Nebulizer
Argon Line
Injector Tip
Auxiliary Argon Line
O-Ring
Coolant Line
Spray Chamber
Drain Tube
Sample Tubing
Support Block

Figure 2



Note: Convex lens should be installed with larger curvature away from the torch.

Operation of the Thermo Jarrell Ash ICAP™ 61E Trace Analyzer Spectrometer

Reference:

1. ICAP™ 61E Trace Analyzer Operator's Manual, July 1993.

Cross Reference:

Document	Document Title
SOP-IO-014	Quality Control Procedure for ICP

Purpose:

The purpose of this SOP is to outline proper operation of the Thermo Jarrell Ash (TJA) ICAP™ 61E Trace Analyzer Spectrometer.

Scope:

This SOP will cover the hardware, software, and quality assurance necessary in the operation of the TJA ICAP™ 61E Trace Analyzer Spectrometer.

Basic Principles:

Water and soil samples are treated with acids and heated to solubilize the metals present. These digestates are then analyzed for trace metals by an atomic emission optical spectroscopic technique. Samples are transported to a nebulizer via an autosampler and peristaltic pump. The nebulizer introduces an aerosol into a spray chamber; the resulting mist is then transported to an argon plasma torch where excitation of atoms occurs. Characteristic atomic-line emission spectra are produced by

a radio-frequency (R.F.) inductively coupled plasma. The spectra are dispersed by a diffraction grating and the intensities of the light at each wavelength are monitored by a series of photosensitive devices. The signals from the photosensitive devices are processed by a computer. A background correction technique is required to compensate for variable background contribution to the spectra of trace elements.

Personnel Training and Qualifications:

1. Review and understanding of this procedure.
2. Trainee observing trained analyst performing the procedure.
3. Trainer observing trainee performing the procedure.
4. Review of the trainee's data by trainer.
5. Documentation of critical steps in the training process.
6. Demonstration of proficiency by being able to independently run the ICP.

Interferences:

Spectral interferences are caused by background emission, stray light from high concentration elements or overlap from a spectral line from another element. Spectral interferences can be compensated for by the use of background points, alternate wavelengths and interelement corrections.

Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for by using internal standardization.

Memory interference, or carryover, is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time of the autosampler tubing overcomes any memory interference.

Apparatus and Equipment:

The following is a list of the hardware used in the TJA ICAP™ 61E Trace Analyzer Spectrometer systems. More detailed information can be obtained the from the *Operator's Manual.*

1. Spectrometer – The TJA ICAP™ 61E Trace Analyzer is a simultaneous plasma-emission spectrometer. It consists of a 0.75 M Rowland Circle; Paschen-Runge mount polychromator under vacuum, an R.F. generator, axially mounted inductively coupled argon plasma, a 2400-grooves/mm grating, and a data acquisition system.

2. Autosampler – The Cetac SX-510 Autosampler has a capacity for 240 samples and 10 standards, and has one rinse reservoir. The parameters for each automated run are entered into the autosampler table in the ICP Manager™ software as described in Section 3 of the Procedure in this SOP.

3. Peristaltic pump – The peristaltic pump regulates the flow of the following: sample, internal standard, instrument rinse and spray chamber waste. Special care must be taken to ensure that all pump tubing is connected properly. The Meinhard Type K Nebulizer has a natural uptake, but an external peristaltic pump is used to compensate for differences in sample viscosity. After travelling through the peristaltic pump, the sample and internal standard tubing are combined by a "Y" connector, and then allowed to mix in a mixing coil before entering the nebulizer.

4. R.F. generator – R.F. generator is crystal controlled at 27.12 MHz. The power tube is directly coupled to the induction coil, which allows for efficient transmission of signal between the two.

5. Coolflow – The Thermo Jarrell Ash Coolflow WAC050 is set to deliver cooling water at the rate of 300 mL/min with a minimum pressure of 30 psig and a maximum pressure of 60 psig.

6. Personal computer – The TJA ICAP™ 61E Trace Analyzer is controlled by a PC.

7. Vacuum pump – The Alcatel vacuum pump puts the entire optical system under vacuum. This allows for better viewing of the lower wavelengths and less sensitivity to environmental changes.

Procedure:

The TJA ICAP™ 61E Trace Analyzer is operated on ICP Manager™ software

1. From the Start menu, select "ICP Manager" to start the software

2. Warm start-up of TJA ICAP™ 61E Trace Analyzer

   a. Check whether or not the standby and fatigue lights are lit. If they are not, press the Reset button on the power distribution panel on the back of the instrument and the Reset button on the front of the instrument. If these lights are not lit after resetting the instrument, verify that the high voltage power switch is on. If the high voltage power switch is off, then perform a cold start. (See Section 10 of the *ICAP™ 61E Trace Analyzer Operator's Manual.*)

   b. Verify that the vacuum by-pass light is off. If it is on, make sure the vacuum power is on. If it is off, turn it on and press the Start button. The vacuum pressure must be below 30 microns of mercury or the instrument will not function. Check the *Operator's Manual*, Section 11, if correct vacuum pressure cannot be obtained.

   c. Ensure that the drain tubing for the spray chamber is properly connected to the peristaltic pump and positioned to drain into a waste carboy.

   d. Verify that all other tubing is connected properly. The peristaltic pump should be off.

e. Access the Torch Ignition window in the instrument software by selecting "Ignite plasma" from the "Instrument" menu.

f. In the Torch Ignition window, select "Ignite."

g. After the plasma has ignited, turn the peristaltic pump on (it should be turning clockwise). If the plasma does not light, repeat steps e and f. If plasma still does not light, repeat steps a-f and/or refer to Section 19 of the Operator's Manual.

h. Once the plasma operating parameters have engaged, exit the Torch Ignition window by clicking on Close.

i. If the plasma has been off for more than 15 minutes let the instrument warm up for 30 minutes. If the plasma has been off less than 15 minutes, let the instrument warm up 5 to 10 minutes. The sipper probe should either be at the rinse station or in DI water while the instrument is not in use.

Diagnostic tests to determine and isolate hardware problems are supplied in Section 19 of the *Operator's Manual.*

3. Entering an autosampler table in the ICP Manager™ software

a. Select "Samples" from the menu options, then choose "Sample list editor". This will open the "Sample list editor" window.

b. From the "Sample list editor" File menu, select Open and choose an appropriate template for the run to be typed.

NOTE: TRACE method is used routinely and analyzes 27 analytes using a standard curve generated with a blank and one standard. FAST method functions similarly to TRACE method but does not analyze Be, K, and Tl. By

eliminating these elements from the analysis routine, the spectrum shifter needs to move to fewer points, thus reducing analysis time by approximately 1 minute/tube.

c. Place the cursor between the pre-typed standard groupings and click on the "Run an unknown" icon. The "Analyse unknown" window will appear.

d. Under the "Sample" tab, enter sample number or standard name in the "Sample identity 1" field.

e. For samples, enter the following information in the "Sample identity 2" field with a "/" between each parameter: class, dilution factor, batch number and protocol.

f. After completing these fields, place the information into the autosampler table either by clicking "Insert" or by typing Enter.

g. Repeat steps d-f until all necessary samples and standards have been entered.

h. Click "Continue" to exit the "Analyse unknown" window.

i. Select all lines, then click on the "Resequence..." icon.

j. Change the "New starting tray position" to the appropriate location and click on "Re-sequence".

k. Click on the "Check sequence..." icon to check the sample list for syntax errors. Make corrections and repeat as needed until no errors are found, then click OK.

l. Verify that the sample list begins and ends at the correct tube numbers, and check all entries for errors.

m. If the autosampler table is not for intended for immediate use, proceed to step p in this section; otherwise, edit the Result file name to match the run number.

n. Click on "Develop" from the menus at the top of the screen and select "Method explorer." The "Method explorer" window will appear.

o. Click on the appropriate method name, and enter the necessary rinse time (**NOTE:** The rinse time entered will be in addition to an automatic 35 second rinse performed by the software). Click Close to exit the "Method explorer" window.

p. From the "Sample list editor" File menu, select Save as. Name the file as the first batch on the run.

q. Click on the Print icon to print the autosampler list. This printout should be kept with the run cover sheet.

r. Click Close to exit the "Sample list editor" window.

4. Profiling in ICP Manager™

It is required that the TJA ICAP™ 61E Trace Analyzer be profiled every 8 hours at a minimum. More frequent profiling is recommended.

a. Select "Automated profile" from the Instrument menu in the ICP Manager. software.

b. Select the As line by clicking in the box next to As. Make sure that a checkmark appears in the box.

c. If using the autosampler, click on the box next to "Use autosampler positioning" and adjust the "Tray position" to the location of the Arsenic Profile Solution; otherwise, place the sampler probe directly in the solution, being sure to wipe it as it exits the rinse station. Click Next.

d. Click Profile. The instrument will allow 45 seconds of transport time and then begin profiling. If the transport time is not needed, click "bypass" to begin the profile.

e. When the profile has finished, a calculated peak position will appear. The peak position should be within ±0.1 of 0. If it is not, change the Vernier position on the front panel of the instrument to the New Vernier position calculated by the software. Click on the save icon to save changes before exiting and then start a new profile from Step a of this section.

f. If the peak position is acceptable, click on the print icon to open the Print preview window and record all necessary information in the instrument run log. Close the Print preview window by clicking Close.

g. Click Cancel to exit the automated profile without saving changes.

h. The autosampler will automatically return the sampler probe to the rinse station, but if the autosampler was not used the probe must be rinsed in DI water, wiped, and then returned to the autosampler arm.

5. Autosampler analysis in ICP Manager™

a. If an autosampler table has already been created for the run to be started, select "Samples" from the menu options, then choose "Sample list editor". This will open the "Sample list editor" window. To create an autosampler table, refer to Section 3 of the Procedure in this SOP.

b. From the "Sample list editor" File menu, select Open and select the appropriate file.

c. Make sure that the Result file name matches the run number.

d. Verify all information is accurate, and that the correct method is used.

e. If changes have been made, click on the Print icon to print the autosampler list. This printout should be kept with the run cover sheet.

f. To change the rinse time, click on "Develop" from the menus at the top of the screen and select "Method explorer". The "Method explorer" window will appear. Click on the appropriate method name, and enter the necessary rinse time (NOTE: The rinse time entered will be in addition to an automatic 35 second rinse performed by the software). Click Close to exit the "Method explorer" window. This step ensures that the current method information will be used.

g. Click the Queue icon to add the current sample file to the run queue. Choose Yes to replace the existing file. The Autosampler analysis window will automatically open.

h. Change the view to the "Current sample list" view and click on the "Drift standards" tab to verify that drift correction standards will be run.

i. Click on the green circle to begin autosampler analysis.

6. Manual analysis in ICP Manager

a. Select "Manual analysis" from the Analysis menu to open the Manual analysis window.

b. Enter all necessary information in the Sample identification fields. Sample type should be "Unkown."

c. Select "Use autosampler" and change the Tray position to the appropriate location.

d. Ensure that the method to be used matches that of the last run on the instrument.

e. Click on the green circle to begin manual analysis.

f. After analysis is complete, click No when asked to save data.

g. Click on the Print icon if necessary.

h. Click Close to exit the Manual analysis window.

7. Importing run data

a. Start the Parallax LIMS software and log in.

b. Select Import from the IDAT pull-down menu.

c. Open the appropriate run file.

d. Verify that the following information is accurate: sample number, standard name, class, batch number, matrix, protocol, method reference, LCS ID, initial volume, final volume and dilution factor. Make corrections as needed.

e. Enter the correct rinse time.

f. Enter the appropriate analyst number.

g. Click on Check/Save.

h. When the confirmation screen appears, verify that the file name and run number are the same. Choose to either Exit or Import another run and the run will be imported and printed.

8. After import, the data is then reviewed for quality assurance (per SOP-IO-014), uploaded, and verified.

9. Performing an interelement correction (IEC) in ICP Manager™

   a. Prepare a solution of the interfering element at the linear range of the instrument. Perform manual analysis according to section 6 of the Procedure in this SOP.

   b. Note results of elements with a known interference that are greater than the limit of quantitation.

   c. Divide the result of each interfered element by that of the interfering element. These values represent the amount the current IECs need to be adjusted.

   d. Select "Method explorer" from the Develop menu. Expand the list of options for both methods, and select "Interference calibration" for the method used for analysis.

   e. Add each calculated result to the correction factors currently in the C1 column corresponding to each pair of elements. Enter the new correction factors in the table.

   f. Transfer information to the "Interference calibration" tables for the other method.

Revision Log:

Ver. #	Effective Date	Change
00	05/06/96	Previous issue
01	02/26/99	Major changes are as follows: • Added Personnel Training and Qualifications section • Made a few clarifications • Changed from SOP-IO-030 to MC-IO-018
02	04/14/03	Major changes: • Additional clarifications made, including section 5.a • Added macro information in section 3.f • Added "FAST" Method
03	08/14/03	Major changes are as follows: • Section 4.g – Changed the profile solution from Hg to As • Added basic principles • Added interferences • Removed references to "Multi-point" Method
04	09/12/05	Major changes are as follows: • Incorporated Procedural Amendments 1 & 2
05	NOV 0 3 2005	Major changes are as follows: • Apparatus and Equipment section revised to update devices • Procedure section 1-5, 7 and 9 revised to reflect software change; section 6 added

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Prepared by: John A. Wood Date: 9/30/05
Chemist

Approved by: [signature] Date: 10/17/05
Metals Management

Approved by: Elaine Stoltzfus Date: 10/20/05
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Uncontrolled Copy

SOP-IO-005.10
Supersedes Date: 02/14/06
Effective Date: SEP 0 5 2006

Quality Control Procedures for Mercury

Approvals:

Prepared by: _____ Date: 8/15/06
Senior Specialist

Approved by: _____ Date: 8.15.06
Metals Management

Approved by: Dorothy M Love Date: 8/22/06
Quality Assurance

Revision Log:

Ver. #	Effective Date	Change
00	09/19/96	Previous issue
01	08/08/97	Major changes are as follows: • Procedure A - Added statement for SW-846 batches to contain both an analytical spike and serial dilution. • Procedure A.2. - Changed LCS recovery limits for EPA600 to ±15%. • Procedure A.3. - Corrected error in equation concerning relative percent difference. • Procedure A.4. - Separated equations for calculating concentration of spike added for water and soils, and added note for Wisconsin samples. • Procedure B and QC Summary sections. - Added statement saying EPA-600 200.9 has 5% windows for the lst CCV on the run. • Personnel Training and Qualifications section added.
02	02/12/98	Major changes are as follows: • Definitions - Added environmental lead definition • Procedure - Changed blank evaluation to absolute value of the analyte concentration, added HUD Pb requirements • QC Summary - Added absolute value to preparation blank; added HUD Pb requirements to preparation blank, duplicate, initial calibration blank/continuing calibration blank, continuing calibration verification

Ver. #	Effective Date	Change
03	02/25/99	Major changes are as follows: • Additions to Procedure and QC Summary sections are for clarification • Delete "Hydrides" from Procedure section A. • Delete "HUD Pb" from Procedure section A.2. • Change CCV to ICV; delete "the remainder of" in section B. of Procedure • Delete Environmental Lead section • Delete section E. of Procedure • Delete all "HUD" references in QC summary • Add ±5% for ICV EPA 600 200.9; delete ±5% of 1st CCV
04	01/05/00	Major changes are as follows: • Scope – Added mercury. • Procedure B. – Added EPA 600 comment. • QC Summary – Added EPA 600 Comment to (ICV).
05	05/16/01	Major changes are as follows: • Cross Reference section added • QC Summary and Procedure sections – Added description of statistical windows • Changed procedure Title • Changed references to Department 23 to Department 22
06	03/17/03	Major changes are as follows: • Added Reference section • Added CLP 5.2 requirement for CRA/CRI definition and Limits of Quantitation • Added analytical sample Definition • Added LCS limits and CRI limits to Procedure section
07	03/20/03	Major changes are as follows: • Revised entire procedure

Ver. #	Effective Date	Change
08	01/19/05	Major changes are as follows: • Updated Definitions section
09	02/14/06	Major changes are as follows: • Updated Cross Reference and Procedure sections
10	SEP 05 2006	Major changes are as follows: • Updated Procedure A.4, B.1, B.2, B.3, B.4, and the tables

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081506

Reference:

1. Method 7470A (waters) and 7471A (solids), *Test Methods for Evaluating Solid Waste*, USEPA SW-846, September 1994, modified.

2. USEPA CLP SOW No. ILM04.0, Exhibit D/Mercury, CLP-M, modified.

3. Method 245.1, *Methods for Analysis of Water and Wastes*, USEPA 600/4-79-020, Rev. March 1983.

4. Method 245.1, *Methods for the Determination of Metals in Environmental Samples*, Supplement I, EPA-600/R-94/111, May 1994.

5. USEPA CLP SOW No. ILMO5.2, *Exhibit D/Mercury*, CLP-M, modified.

Cross Reference:

Document	Document Title
LOM-SOP-ES-222	Instrument and Equipment Maintenance and Calibration
LOM-SOP-ES-207	Establishing Control Limits
SOP-IO-007, Section E	Mercury Solutions
SOP-IO-007, Section G	Prep Room Solutions (Waters)
SOP-IO-007, Section H	Prep Room Solutions (Solids)
SOP-IO-012	Calculations Used by the Inorganics Group

Purpose:

The purpose of these quality control activities is to provide the means of assuring and maintaining the quality of results generated by Department 22 of Lancaster Laboratories, Inc. In addition, the quality control activities are designed to meet the quality control requirements that are established by various regulatory agencies.

Scope:

This SOP describes the routine quality control for mercury analysis performed by Department 22.

Definitions:

Batch and instrument QC

1. Analytical Samples – Analytical sample is defined as any solution introduced into an instrument on which an analysis is performed, excluding instrument calibration, ICV, ICB, CCV, CCB, and tunes. Analytical samples include undiluted and diluted samples, matrix spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, ICSs, CRIs, LLCs, PBs, LCSs, PEs, and Linear Range Samples (LRSs).

2. Analytical Batch – A group of field samples that are digested and analyzed together. A batch consists of no more than 10 samples for EPA 600 methods or no more than 20 samples for other methods.

3. Background Sample (U) – The original sample from which the batch QC is derived. The background sample is either site specific or randomly selected.

4. Continuing Calibration Blank (CCB) – A reagent blank run immediately after every CCV. This is used to monitor the stability of the low end of the calibration.

5. Continuing Calibration Verification (CCV) – A mid-range standard run at a frequency of 10% (every ten samples) throughout the run. This is used to monitor instrument drift.

6. Contract Required Detection Limit (CRA, CRI) – A standard analyzed at the Contract Laboratory Program (CLP) required detection limit. This standard must be at the beginning of each sample analysis run, but not before the ICV/ICB. This sample verifies linearity near the limit of quantitation. ILM05.2, requires the check standard (now called a CRI) to be analyzed every twenty analytical samples.

7. Duplicate Sample (D) – A replicate of the original sample, processed in parallel. This sample is used to provide a measure of the in-lab repeatability (precision) of the analytical process. The duplicate sample is either site specific or randomly selected.

8. Initial Calibration Blank (ICB) – This is a standard reagent blank, used to prove that the low end of the calibration is acceptable. It must be run immediately after the ICV.

9. Initial Calibration Verification (ICV) – This is a standard near the middle of the calibration range, prepared from a different source than the calibration standards. It is used to prove that the instrument is calibrated correctly at the start of the run.

10. Instrument Detection Limit (IDL) – A value determined from analyzing 7 standard solutions (undigested) at a concentration 3x to 5x the anticipated IDL on three nonconsecutive days. The standard deviation obtained for these multiplied by 3 is the IDL. These must be performed quarterly on each instrument used for an analyte. For non-CLP Analyses – A value determined for the purpose of evaluating the ICB/CCBs for data package samples. It is determined by analyzing 7 standard solutions at a concentration 3x to 5x the anticipated IDL. This value is obtained annually for each element analyzed on an instrument.

11. Laboratory Control Sample (LCS) – This is a matrix-matched synthetic sample of known composition. It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes.

12. Laboratory Control Sample Duplicate (LCSD) – This is a duplicate of the *matrix-matched synthetic sample of known composition.* It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes. It is also used as a measure of the precision of the analytical process.

13. Limits of Quantitation (LOQ) – The level above which quantitative results may be obtained with a specified degree of confidence. It is based on a value 3x to 5x the MDL. CLP 4.0 samples are reported using the IDL and CRDL. CLP 5.2 samples are reported using the MDL and CRQL; the statement of work for the programs specifies required limits.

14. Matrix Spike Sample (R) – A replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure. The matrix spike sample is either site specific or randomly selected.

15. Matrix Spike Duplicate (MSD) – A duplicate of the Matrix Spike Sample (R) which is a replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure. It is also used as a measure of the precision of the analytical process. The matrix spike duplicate sample is either site specific or randomly selected.

16. Method Detection Limit (MDL) – The minimum concentration of a substance that can be reported with 99% confidence that the analyte concentration is greater than 0. It is determined by analyzing 7 digested standards at an estimated concentration 2.5x to 5x the signal/noise ratio. MDLs are performed on all instruments used to determine each analyte.

17. Post Digestion Spike (PDS) – This sample is a spike of the Background Sample prepared after digestion, at the time of analysis. It is used to determine if low spike recoveries are due to problems in the digestion or are *matrix related.*

18. Preparation Blank (PB) – This is a reagent blank carried through the entire digestion procedure. It is used to determine if contamination has occurred during the digestion procedure.

19. Serial Dilution (SD) – This sample is a 1:4 (5x) dilution of the Background *Sample, prepared after the digestion. It is used to indicate the presence of* any matrix effects that could cause a nonlinear response at the instrument.

20. Linear Dynamic Range (LDR) – This is the calibration range for the analyte. The LDR is evaluated on every run. The correlation coefficient for the curve must be ≥0.995.

Personnel Training and Qualifications:

1. Review and understanding of this procedure

2. Trainee observing trained analyst performing the procedure

3. Trainer observing trainee performing the procedure

4. Review of the trainee's data by trainer

5. Documentation of critical steps in the training process

6. Demonstration of proficiency by being able to independently review Hg data

Procedure:

A. Raw data quality checks

1. Make sure that the run is correctly labeled, dated, and signed and that the corresponding cover sheet is attached to the front of the run.

2. For calculations used by the inorganics groups, see SOP-IO-012.

3. For run and batch QC frequency, acceptance criteria and corrective action see Tables I, II, and III. For information on statistical limits see LOM-SOP-ES-207.

4. Each analytical run will have a QC review attached. All samples on the run will be listed on the QC review as to whether the sample was verified or needed to be redigested/reanalyzed. The verifier will document on the QC review if any sample(s) were selected/deselected.

5. For spike levels of run QC, see SOP-IO-007, Section E.

6. For spike levels of batch QC, see SOP-IO-007, Sections G and H.

7. LOQs are available to analysts in the LIMS and on charts that are updated as needed.

8. Check to make sure that all results are within the calibrations range. If a sample reading is above the calibration range, then reread the sample at an appropriate dilution. For CLP 5.2, the diluted sample reading must fall within the upper half of the calibration range.

9. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings ≤LOQ.

10. For TCLP and SPLP samples, an MSA (method of standard additions) is required if the sample concentration falls between 80% to 100% of the regulatory limits.

11. For all EW samples (samples from public drinking water sources), check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once so that the supplier can be notified. Suppliers must be notified within 24 hours.

Analyte	MCL (mg/L)
Hg	0.002

B. When complete, check the following:

1. The beginning of the raw data and QC review are signed and dated by the reviewer/verifier.

2. All samples requiring redigestion are listed on the redigestion schedule forms.

3. Redigest request forms are clipped to the front of the run.

4. The data are uploaded to Parallax via IDAT by reviewer or verified from Parallax by a verifier.

5. The raw data packet is placed in the verification bin. (For samples following Good Laboratory Practices [GLP], the raw data includes the "real-time" printout, as well as the final print file. The "real-time" printout should be signed and dated by the analyst).

C. Instrument and method detection limits are performed on each analytical instrument on a yearly basis. In addition, instruments used for the Contract Laboratory Program under CLP SOW ILM04.0 must have instrument detection limits on a quarterly basis for those required elements. These detection limit checks assist in the identification of potential instrument problems and assure that the reported quantitation limits are obtainable.

D. Taking an instrument/analysis out of service/returning an instrument/analysis to service

NOTE: The following is taken from LOM-SOP-ES-222. In the event of an equipment failure, the following shall be performed:

1. Document the nature of the failure in the maintenance logbook

2. Document how and when the defect was discovered

3. Notification of supervisor or responsible person who can decide on appropriate action to take

4. The instrument must be clearly tagged as *Out of Service*. The tag must contain the following information:

    a. Date taken out of service

    b. Employee who took the instrument out of service

    c. Reason for tagout

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken to bring the equipment back into service.

7. Results of the corrective action (i.e., system calibration within specifications, etc.)

8. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument. This will be done on a case-by-case basis.

9. After repair, document whether the function has been fixed. Calibration or verification activities may need to be performed before the instrumentation is put back into service.

Table I

QC requirements for CLP 4.0 and 5.2.
(Mercury)

	Frequency	Acceptance	Corrective Action
Calibration	The calibration will contain a blank and 5 standards	Correlation coefficient > 0.995	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Verification (ICV)	Must be analyzed immediately following the calibration.	± 10% of the True Value	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV	Must be < | CRDL (ILMO4.0) Must be < | CRQL (ILMO5.2)	Data for that analyte cannot be reported from the run (reanalyze).
Contract Required Detection Limit (CRA)	Must be analyzed immediately after the ICB (ILMO4.0) Must be analyzed at the beginning of the run immediately after the ICB, at the end of the run before the final CCV and after every 20 analytical samples (ILMO5.2)	± 50% of the True Value (ILMO4.0) ± 30% of the true value (ILMO5.2)	Data for that analyte cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the CRA and at a frequency of every 10 samples	± 20% of the true value	Data bracketing the CCV for that analyte cannot be reported from the run (reanalyze). If the CCV is out of specification, it can be read in duplicate. If both CCVs are within specification, the data from the last good CCV may be reanalyzed. If one or both CCVs are still out of specification, then the run will be terminated and the samples after the last good CCV will need to be reanalyzed on a new run.
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every ten samples	Must be < | CRDL (ILMO4.0) Must be < | CRQL (ILMO5.2)	Data bracketing the CCB for that analyte cannot be reported from the run (reanalyze). If the CCB is out of specification, it can be read in duplicate. If both CCBs are within specification, the data from the last good CCB may be reanalyzed. If one or both CCBs are still out of specification, then the run will be terminated and the samples after the last good CCB will need to be reanalyzed on a new run.
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Must be < | CRDL (ILMO4.0) Not applicable if analyte	Redigest all associated samples.

Table I – Continued

QC requirements for CLP 4.0 and 5.2. (Mercury)			
	Frequency	Acceptance	Corrective Action
		reading in the sample is > 10x the PB reading or < CRDL/CRQL	
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits for solid matrix Use ± 20% of True Value for water matrix If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC than a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less	Use statistical limits for solid matrix Use ± 20% of True Value for water matrix If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ.
Matrix Spike (MS)	Must be prepped of a frequency of 1 per analytical batch of 20 samples or less	Use statistical limits or the method limit of ± 25% whichever is tighter	The data is flagged in the QC Summary and/or in the data package.
Duplicate (D)	Must be prepped of a frequency of 1 per analytical batch of 20 samples or less	If the samples are > 5x the CRLD/CRQL the RPD must be < 20. If either the sample or duplicate is < 5x the CRDL/CRQL the difference between the two values must be < CRDL/CRQL. Not applicable if both samples are < CRDL/CRQL.	The data is flagged in the QC Summary and/or in the data package.

Table II

QC requirements for EPA600 245.1 (Mercury) for (PW, EW) and (WW)

	Frequency	Acceptance	Corrective Action
Calibration	The calibration will contain a blank and 5 standards	Correlation coefficient > 0.995	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Verification (ICV)	Must be analyzed immediately following the calibration.	±10% of the True Value (WW) ±5% of the True Value (PW, EW)	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV	Must be < |LOQ|.	Data for that analyte cannot be reported from the run (reanalyze).
Contract Required Detection Limit (CRA)	Must be analyzed immediately after the ICB	± 50% of the True Value.	Data for that analyte cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the CRA and at a frequency of every 10 samples	± 10% of the true value	Data bracketing the CCV for that analyte cannot be reported from the run (reanalyze). If the CCV is out of specification, it can be read in duplicate. If both CCVs are within specification, the data from the last good CCV may be reanalyzed. If one or both CCVs are still out of specification, then the run will be terminated and the samples after the last good CCV will need to be reanalyzed on a new run.
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every ten samples	Must be < |LOQ|.	Data bracketing the CCB for that analyte cannot be reported from the run (reanalyze). If the CCB is out of specification, it can be read in duplicate. If both CCBs are within specification, the data from the last good CCB may be reanalyzed. If one or both CCBs are still out of specification, then the run will be terminated and the samples after the last good CCB will need to be reanalyzed on a new run.
Preparation Blank (PB)	Must be prepped at a frequency of	Must be < |LOQ|.	Redigest all associated samples.

Table II – Continued

QC requirements for EPA600 245.1 (Mercury) for (PW, EW) and (WW)			
	Frequency	Acceptance	Corrective Action
	1 per analytical batch of 10 samples or less.	Not applicable if analyte reading in the sample is > 10x the PB reading or <LOQ.	
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	Use statistical limits or ± 15%whichever is tighter If the LCS is out of specification high and the samples are less than the LOQ the data can be taken	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 10 samples or less	Use statistical limits or ± 15%whichever is tighter If the LCS is out of specification high and the samples are less than the LOQ the data can be taken	Redigest all associated samples if the LCS is out of specification low If the LCS is out of specification high redigest samples that are greater than the LOQ
Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less	Use statistical limits or the method limit of ± 30% (PW,EW) / ± 20% (WW) whichever is tighter Not applicable if sample concentration is > 4x spike added.	The data is flagged in the QC Summary and/or in the data package.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less	If the samples are > 5x the LOQ the RPD must be < 20. If either the sample or duplicate is < 5x the LOQ the difference between the two values must be < LOQ. Not applicable if both samples are < LOQ.	The data is flagged in the QC Summary and/or in the data package.

Table III

QC requirements for EPA SW-846 7470A and 7471A (Mercury)

	Frequency	Acceptance	Corrective Action
Calibration	The calibration will contain a blank and 5 standards	Correlation coefficient >0.995	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Verification (ICV)	Must be analyzed immediately following the calibration.	±10% of the True Value	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV	Must be < |LOQ|	Data for that analyte cannot be reported from the run (reanalyze).
Contract Required Detection Limit (CRA)	Must be analyzed immediately after the ICB	± 50% of the true value.	Data for that analyte cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the CRA and at a frequency of every 10 samples	± 20% of the true value	Data bracketing the CCV for that analyte cannot be reported from the run (reanalyze). If the CCV is out of specification, it can be read in duplicate. If both CCVs are within specification, the data from the last good CCV may be reanalyzed. If one or both CCVs are still out of specification, then the run will be terminated and the samples after the last good CCV will need to be reanalyzed on a new run.
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every ten samples	Must be < |LOQ|	Data bracketing the CCB for that analyte cannot be reported from the run (reanalyze). If the CCB is out of specification, it can be read in duplicate. If both CCBs are within specification, the data from the last good CCB may be reanalyzed. If one or both CCBs are still out of specification, then the run will be terminated and the samples after the last good CCB will need to be reanalyzed on a new run.
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20	Must be < |LOQ|. Not applicable if analyte	Redigest all associated samples.

Table III – Continued

QC requirements for EPA SW-846 7470A and 7471A (Mercury)			
	Frequency	Acceptance	Corrective Action
	samples or less.	reading in the sample is > 20X the PB reading or < LOQ.	
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or ± 20%whichever is tighter If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken	Rediges*t all associated samples if the* LCS is out of specification low. If the LCS is out of specification high *rediges*t *samples that are* greater than the LOQ.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less	Use statistical limits or ± 20%whichever is tighter If the LCSA is out of specification high and the sample result is less than the LOQ the data can be taken	Rediges*t* all associated samples if the LCS is out of specification low. If the LCS is out of specification high rediges*t* samples that are greater than the LOQ.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less	Use statistical limits or the method limit of ± 20% whichever is tighter. Not applicable if sample concentration is > 4X spike added.	*The data is flagged in the QC* Summary and/or in the data package.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less	*If the samples are* > 5X the LOQ the RPD must be < 20. If either the sample or duplicate is < 5X the LOQ the difference between the two values must be < LOQ. Not applicable if both samples are < LOQ.	*The data is flagged in the QC* Summary and/or in the data package.
Post Digestion Spike (PDS)	Prepared with each solid background sample. Evaluated when matrix spike/spikes are not within specification.	± 25% of the true value.	The data is reported in the data package.

Table III – Continued

QC requirements for EPA SW-846 7470A and 7471A (Mercury)			
	Frequency	Acceptance	Corrective Action
Serial Dilution	Prepared with each solid background sample. Evaluated only when analyte concentrations are > 50X the MDL	The percent difference must be < 10%	The data is flagged in the data package.

Preparation of Standards and Solutions

Purpose:

The following procedures should be used to prepare standards and solutions to be used in analysis for various metals. Unless otherwise stated:

1. Intermediate and general standard solutions holding time listed with individual preparation instructions.

2. Calibration standards have a 1-month holding time.

3. Reference to acid is concentrated acid.

4. Reference to water is deionized water.

5. Storage containers are polyethylene bottles.

All standards and reagents must be labeled with the solution name, lot number, date prepared, the expiration date, the initials of the person preparing the solution, and the storage conditions.

If larger or smaller volumes are needed, adjust all additions according to final solution volume.

After diluting to volume, all solutions are thoroughly mixed.

Storage conditions for all reagents and solutions is room temperature.

NOTE: All new lots of acid used for solution preparation are first evaluated at a 1:10 dilution by trace ICP. All analytes must be less than the water MDL for that element. Acids that have been certified by the manufacturer to those levels are also acceptable.

Scope:

The procedure is a listing of all reagents prepared in department 22.

Personnel Training and Qualifications:

All personnel responsible for making standards will have a thorough knowledge of this written procedure. A trainee will first observe a trained analyst perform the task and then be observed by a trained analyst as he/she performs the task. General laboratory safety training will be obtained in accordance to the Lancaster Laboratories Chemical Hygiene Plan.

Contents:

A. Reagents

B. Atomic absorption solutions – Analysis no longer run

C. ICP Solutions

C.1 ICP standards

C.1.1 ICP Calibration Blank Solution (S0)

C.1.2 ICP Calibration Standard 1 (S1)

C.1.3 ICP Calibration Standard 2 (S2)

C.1.4 ICP Calibration Standard 3 (S3)

C.2 ICP Initial Calibration Verification (ICV)

C.3 ICP Continuing Calibration Verification (CCV)

C.4 ICP Initial and Continuing Calibration Blank (ICB/CCB)

C.5 ICP LOQ/CRDL/CRQL Check Standard Solutions (CRI, LLC)

C.5.1 Low Level Check (LLC)

C.5.2 CLP 2.1, CLP 4.0 CRI

C.5.3 CG CRI Intermediate Solution (CG CRI INT)

C.5.4 CG CRI working solution

C.5.5 CLP 5.2 CRI

C.5.6 Texas Risk Reduction Program (TRRP) CRI

C.6 Interference Check Solutions

C.6.1 ICP Interferent Check Solution A (ICSA)

C.6.2 ICP Interferent Check Solution AB (ICSAB)

C.7 ICP Rinse Solutions

C.7.1 ICP Rinse Solution for dilutions

C.7.2 ICP Rinse Solution for instrument

C.8 Arsenic (1-ppm) Profile Solution

C.9 Y-Lithium (10-ppm)/Lithium (1000-ppm) Internal Standard

C.10 ICP Instrument Detection Limit (IDL) stock solution

C.11 ICP Method Detection Limit (MDL) stock solution

C.12 ICP Calibration Standards

C.12.1 Calibration Standard 4

C.12.2 Calibration Standard 5

C.12.3 Calibration Standard 6

C.12.4 Calibration Standard 7

C.12.5 Calibration Standard 8

C.13.6 Calibration Standard 9

C.13.7 Calibration Standard 10

C.13.8 Calibration Standard 11

C.13.9 Calibration Standard 12

C.13.10 Calibration Standard 13

C.13.11 Calibration Standard 14

D. ICPMS solutions

D.1 ICPMS instrument rinse

D.2 ICPMS ICB/CCB/CCS/Rinse

D.3 ICPMS calibration blank solution (S0)

D.4 ICPMS calibration standard 1 (S1)

D.5 ICPMS ICV

D.6 ICPMS CCV

D.7 Low level check solutions

D.7.1 Low Level Check (LLC)

D.7.2 Drinking Water – Be/Tl Low Level Standard

D.7.3 Contract Required Quantitation Limit Check (CRI)

D.8 ICPMS interference check solutions

D.8.1 ICPMS ICSA solution

D.8.2 ICPMS ICSAB solution

D.9 Internal standard

D.10 Tuning solution

D.11 Dual detector calibration solution

D.12 Auto lens calibration solution

D.13 Non-CLP PDS Spike

D.14 ICPMS LRS solution

E. Mercury solutions

E.1 Intermediate calibration standards

E.1.1 Intermediate (10 mg/L)

E.1.2 Intermediate (1.0 mg/L)

E.1.3 Intermediate (0.1 mg/L)

E.2 Manual digestion standards

E.2.1 Calibration standards

E.2.2 Control (ICV) (0.0025 mg/L)

E.2.3 Control (ICV) (0.0020 mg/L)

E.2.4 Low level (CRA) (0.0002 mg/L)

E.2.5 Continuing calibration verification (CCV) 0.0010mg/L

E.3 Leeman Labs Hydra Prep digestion standards

E.3.1 1% nitric acid soln.

E.3.2 Calibration standards

E.3.3 Control (ICV) (0.0025 mg/L)

E.3.4 Control (ICV) (0.0020 mg/L)

E.3.5 Control (CCV/LCS) (0.0010 mg/L)

E.3.6 Low level (CRA) (0.0002 mg/L)

E.4 General solutions

E.4.1 Potassium permanganate (5%)

E.4.2 Potassium persulfate (5%)

E.4.3 Sodium chloride/hydroxylamine hydrochloride

E.4.4 Stannous chloride varian (25%) – No longer run

E.4.5 Stannous chloride Leeman Labs (10%)

E.4.6 1% solution nitric acid

F. Graphite furnace solutions

F.1 Calibration standards

F.1.1 Calibration blank

F.1.2 Stock solutions

F.1.3 Beryllium and cadmium intermediate

F.1.4 Calibration stock

F.1.5 Calibration standard

F.1.6 Lead intermediate

F.1.7 Lead calibration stock

F.1.8 Lead calibration standard

F.1.9 Silver calibration stock

F.1.10 Silver calibration standard

F.1.11 Thallium potable water calibration standard

F.2 Control solutions

F.2.1 Initial calibration verification stock (ICV)

F.2.2 Initial calibration verification standard (ICV)

F.2.3 Thallium potable waters initial calibration verification standard (ICV)

F.2.4 Arsenic, antimony, thallium intermediate (CCV)

F.2.5 Beryllium and cadmium intermediate (CCV)

F.2.6 Continuing calibration verification stock (CCV)

F.2.7 Continuing calibration verification standard (CCV)

F.2.8 Silver calibration verification stock

F.2.9 Silver calibration verification standard (CCV)

F.2.10 Thallium potable waters continuing calibration verification standard (CCV)

F.2.11 Contract required detection limit check (CRA)

F.2.12 Lead contract required detection limit check (CRA)

F.2.13 Silver contract required detection limit check (CRA)

F.2.14 Thallium potable waters contract required detection limit check

F.3 Modifiers

F.3.1 Ammonium phosphate – magnesium nitrate

F.3.2 Ammonium phosphate – magnesium nitrate – ascorbic acid

F.3.3 Palladium nitrate

F.3.4 Lanthanum Oxide

F.3.5 Magnesium nitrate – palladium nitrate

F.4. Spike solutions

F.4.1 MSA spike solution

F.4.2 Se spike solution

F.4.3 Tl Potable water MSA spike solution

F.4.4 Silver MSA spike solution

G. Prep room solutions (waters)

G.1 CLP spike solutions

G.1.1 Soln 1 – Ca, Mg, K, Na, Li

G.1.2 Soln 2 – Ag, Be, Cd

G.1.3 Soln 3 – Cr, Cu, Fe, Mn, Ni, Tl, Zn

G.1.4 Soln 4 – Al, Ba, Pb, Mo, Sn, V, B

G.1.5 Soln 5 – Si, Sr, Ti, Co, Sb, As, Se

G.1.6 Soln 6 – Hg

G.1.7 Soln 7 – No longer used

G.1.8 Soln 8 – GF - As, Cd, Pb, Se, Tl, Sb, Cu, Ni, Cr, Be

G.1.9 Soln 10 – No longer used

G.1.10 Soln 11 – Cr, Cu, Fe, Mn, Ni, Tl, Zn, Pb, As, Se

G.1.11 Soln 12 – Al, Ba, Mo, Sn, V, B

G.1.12 Soln 13 – Si, Sr, Ti, Co, Sb

G.2 CLP spike solution addition/theoretical recovery for spiked samples

G.3 Graphite furnace silver spike (also used for solids)

G.4 General solutions

G.4.1 Methyl orange – No longer used

G.4.2 Sodium hydroxide solution (8 g/L) – No longer used

G.4.3 Potassium permanganate (5%) – No longer used

G.4.4 Calcium chloride (15 g/L) No longer used

G.4.5 Nitric acid (1:1)

G.4.6 Hydrochloric acid (1:1)

G.4.7 100 mg/L Cr spike soln.

H. Prep room solutions (solids)

H.1 CLP spike solutions

H.1.1 Soln 1 - Ca, Mg, K, Na, Li

H.1.2 Soln 2 - Ag, Be, Cd

H.1.3 Soln 3 - Cr, Cu, Fe, Mn, Ni, Tl, Zn

H.1.4 Soln 4 - Al, Ba, Pb, Mo, Sn, V, B

H.1.5 Soln 5 - Sr, Ti, Co, Sb, As, Se, Si

H.1.6 Soln 6 – Hg

H.1.7 Soln 7 – No longer used

H.1.8 Soln 8 - GF - As, Cd, Pb, Se, Tl, Sb, Cu, Ni, Cr, Be

H.1.9 Soln 11 – Cr, Cu, Fe, Mn, Ni, Tl, Zn, Pb, As, Se

H.1.10 Soln 12 – Al, Ba, Mo, Sn, V, B

H.1.11 Soln 13 – Si, Sr, Ti, Co, Sb

H.2 CLP spike solutions addition/theoretical recovery for spiked samples

H.3 Graphite furnace silver spike

H.4 General solutions

H.4.1 Acetic acid (4%) – No longer run

H.4.2 Nitric acid, bottle rinse (2N)

H.4.3 Nitric acid (1:1)

H.4.4 Hydrochloric acid (0.07N) – No longer run

H.4.5 Hydrochloric acid (2N) – No longer run

H.4.6 Glassware cleaning soln – No longer run

H.4.7 Aqua regia

H.4.8 Air filter acid cocktail – No longer run

H.4.9 Hydride acid cocktail – No longer run

H.4.10 100 mg/L Cr spike soln.

H.5 Mercury solutions

H.5.1 Potassium permanganate (5%)

H.5.2 Potassium persulfate (5%)

H.5.3 Sodium chloride/hydroxylamine hydrochloride

H.5.4 Stannous chloride (25%) varian – No longer run

H.5.5 Stannous chloride (10%) Leeman Labs PS200II

H.5.6 Mercury intermediate (10 mg/L)

H.5.7 Mercury intermediate (1.0 mg/L)

H.5.8 Mercury intermediate (0.1 mg/L)

H.5.9 Mercury calibration standards

H.5.10 Mercury control (ICV) (0.0025 mg/L)

H.5.11 Mercury continuing calibration (CCV) (0.001 mg/L)

H.5.12 Mercury low-level (CRA) (0.0002 mg/L)

Revision Log:

Ver. #	Effective Date	Change
00	10/16/95	New
01	05/30/00	Major changes are as follows: • Sections deleted – Solutions no longer in use • Incorporated Procedural Amendment #2
02	05/17/01	Major changes are as follows: • Added F.3.5 – Magnesium Nitrate - palladium nitrate • Personnel Training and Qualification – Section added • Typing corrections throughout • Mercury ICV solution changed to (0.0025 mg/L) • Updated Mercury instrument to AP200II and PS200II
03	08/14/03	Major changes are as follows: • Reformatted to Level 3
04	12/19/03	Major changes are as follows • Section D.– Updated index to reflect changes in ICPMS standards section
05	08/17/04	Major changes are as follows: • Change all references of AP200II to Hydra Prep • Insert ICV (0.0020 mg/L) to sections E.2 and E.3 • Added Scope section
06	02/21/05	Major changes are as follows: • Order and identification of standards in Section C changed and updated
07	09/07/05	Major changes are as follows: • Updated index to reflect changes in SOP-IO-007D.

Ver. #	Effective Date	Change
08	NOV 3 0 2005	Major changes are as follows: • Updated index to reflect changes in SOP-IO-007D.

SOPIO007_08.DOC
110805

Prepared by: David F Beck Date: 11/8/05
Chemist

Approved by: Robert Stroeckle Date: 11.14.05
Metals Management

Approved by: Elaine Stoltzfus Date: 11/16/05
Quality Assurance

A. Reagents*

Ammonium phosphate, monobasic crystal ($[NH_4]H_2PO_4$) Baker Analyzed Reagent

Hydrochloric acid (HCl) 36.5% to 38%, Baker Instra-Analyzed Reagent, 1.194 g/mL

Hydrofluoric acid (HF) 48.0% to 51.0%, Baker Analyzed Reagent

Hydrogen peroxide, 30% (H_2O_2) Fisher, Certified A.C.S.

Hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) Fisher, Certified A.C.S.

L-(+)-Ascorbic acid, powder ($C_6H_8O_6$) Baker Analyzed Reagent

Lanthanum oxide (La_2O_3) 99.9+%, Aldrich, AAS Grade

Magnesium nitrate, 6 hydrate, crystal ($Mg[NO_3]_2$) Baker Analyzed Reagent

Nitric acid (HNO_3) 70.0% to 71.0%, Baker Instra-Analyzed Reagent, 1.428 g/mL

Palladium nitrate (1% $Pd[NO_3]_2$) Perkin Elmer

Potassium dichromate ($K_2Cr_2O_7$) Mallinckrodt, primary standard

Potassium permanganate ($KMnO_4$) Baker Analyzed Reagent, A.C.S.

Potassium persulfate ($K_2S_2O_8$) Baker Instra-Analyzed Reagent, A.C.S.

Sodium chloride (NaCl) Fisher, Certified A.C.S.

Stannous chloride ($SnCl_2 \cdot 2H_2O$) Mallinckrodt, AR

Sulfuric acid, 95.0% to 98.0% (H_2SO_4) 36 N, Fisher, Reagent A.C.S., 1.84 g/mL

*Use these or equivalent reagents.

Revision Log:

Ver. #	Effective Date	Change
00	07/11/95	Previous issue
01	08/17/04	Major changes are as follows: • Delete: Acetic acid, Boric acid, Methyl orange indicator, Potassium iodide, and Urea
02	SEP 16 2004	Major changes are as follows: • Updated to Level 3

SOPIO007A_02.DOC
083104

Prepared by: Eugene K. Alicl Date: 9-1-04
Senior Chemist Coordinator

Approved by: Robert Strecker 8/1 Date: 9.1.04
Metals Management

Approved by: Dorothy McKee Date: 9/2/04
Quality Assurance

Basic Principles:

Standards and solutions are prepared for use in analysis for various metals by following the standard operation procedure. Containment vessel is labeled as to content, date of preparation, date of expiration, and storage conditions. The analyst preparing the solution must also label the standard with their initials and employee number as well as documenting the preparation procedure in the Standard Prep Log.

C. ICP Solutions (ICAP™ 61E Trace Analyzer)

All ICP solutions are to be made with ASTM Type II deionized water and Baker Analyzed acids for trace metals analysis, unless otherwise noted. All solutions will be stored at room temperature. The sources listed for the prepared standards solutions are recommended. Equivalent solutions may be used. The standards listed in section C.1 must be from a different source than the remaining standards. All ICP standards are acid-matrix matched to the acid concentration used in the digest (see Figure 1). Final volumes may be adjusted if all the components in the solution are adjusted accordingly.

See LOM-SOP-ES-225 for the appropriate labeling and documentation of reagent and standard preparation.

*Denotes that it is a premixed standard solution. The holding time for all the solutions described is 1 month, unless otherwise noted.

C.1 ICP Standards

C.1.1 ICP Calibration Blank Solution (S0)

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acids according to Figure 1, use Fisher, Trace Metals Grade.

Bring to volume with deionized water.

C.1.2 ICP Calibration Standard 1 (S1)

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acids according to Figure 1.
10 mL TRACE CAL SOL'N #1* (VHG Labs, Inc.)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Al	5000.0	50.0
Ca	5000.0	50.0
Fe	5000.0	50.0
Mg	5000.0	50.0
K	5000.0	50.0
Na	5000.0	50.0

C.1.3 ICP Calibration Standard 2 (S2)

In a 500-mL volumetric flask containing about 250 mL deionized water, add the following:

Add acids according to Figure 1.
2.5 mL TRACE CAL SOL'N #2* (VHG Labs, Inc.)
0.5 mL 1000 ppm Boron
0.5 mL 1000 ppm Strontium

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	200.0	1.0
As	200.0	1.0
B	1000.0	1.0
Ba	200.0	1.0
Be	200.0	1.0
Cd	200.0	1.0
Co	200.0	1.0
Cu	200.0	1.0
Mn	200.0	1.0
Ni	200.0	1.0
Pb	200.0	1.0
Se	200.0	1.0
Sr	1000.0	1.0
Tl	200.0	1.0
Zn	200.0	1.0

C.1.4 ICP Calibration Standard 3 (S3)

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acids according to Figure 1.
5.0 mL TRACE CAL SOL'N #3* (VHG Labs, Inc.)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Cr	200.0	1.0
Mo	200.0	1.0
Sb	200.0	1.0
Sn	200.0	1.0
Ti	200.0	1.0
V	200.0	1.0

C.2 ICP Initial Calibration Verification (ICV)

In a 200-mL volumetric flask containing about 100 mL deionized water, add the following:

Add acids according to Figure 1.
5.0 mL Trace ICV I* (High Purity Standards)
1.0 mL Trace ICV II* (High Purity Standards)

Bring to volume with deionized water. Holding time is 5 days.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	120.0	0.6
Al	1200.0	30.0
As	120.0	0.6
B	120.0	0.6
Ba	120.0	0.6
Be	120.0	0.6
Ca	1200.0	30.0
Cd	120.0	0.6
Co	120.0	0.6
Cr	120.0	0.6
Cu	120.0	0.6
Fe	1200.0	30.0
K	1200.0	30.0
Mg	1200.0	30.0
Mn	120.0	0.6
Mo	120.0	0.6
Na	1200.0	30.0
Ni	120.0	0.6
Pb	120.0	0.6
Sb	120.0	0.6
Se	120.0	0.6
Sn	120.0	0.6
Sr	120.0	0.6
Ti	120.0	0.6
Tl	120.0	0.6
V	120.0	0.6
Zn	120.0	0.6

C.3 ICP Continuing Calibration Verification (CCV)

In a 200-mL volumetric flask containing about 150 mL deionized water, add the following:

Add acids according to Figure 1.
5.0 mL Trace CCV I* (High Purity Standards)
1.0 mL Trace CCV II (High Purity Standards)

Bring to volume with deionized water. Holding time is 5 days.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	100.0	0.5
Al	1000.0	25.0
As	100.0	0.5
B	100.0	0.5
Ba	100.0	0.5
Be	100.0	0.5
Ca	1000.0	25.0
Cd	100.0	0.5
Co	100.0	0.5
Cr	100.0	0.5
Cu	100.0	0.5
Fe	1000.0	25.0
K	1000.0	25.0
Mg	1000.0	25.0
Mn	100.0	0.5
Mo	100.0	0.5
Na	1000.0	25.0
Ni	100.0	0.5
Pb	100.0	0.5
Sb	100.0	0.5
Se	100.0	0.5
Sn	100.0	0.5
Sr	100.0	0.5
Ti	100.0	0.5

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Tl	100.0	0.5
V	100.0	0.5
Zn	100.0	0.5

C.4 ICP Initial and Continuing Calibration Blank (ICB/CCB)

In a 1000-mL volumetric flask containing about 500 mL of deionized water, add the following:

Add acids according to Figure 1.

Bring to volume with deionized water. Mix completely.

C.5 ICP LOQ/CRDL/CRQL Check Standard Solutions (CRI, LLC)

C.5.1 Low Level Check (LLC)

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acid according to Figure 1.
0.2 mL Low Level Check* (SCP Science)
0.2 mL LLC – Sn (SCP Science)
0.2 mL LLC – Sb (SCP Science)

Bring to volume with deionized water. All elements are spiked at the limit of quantitation.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	25	0.005
Al	1000	0.2
As	50	0.01
B	250	0.05
Ba	25	0.005
Be	25	0.005
Ca	1000	0.2
Cd	25	0.005
Co	25	0.005
Cr	25	0.005
Cu	50	0.01
Fe	1000	0.2
K	1000	0.5
Mg	500	0.1
Mn	25	0.005
Mo	50	0.01
Na	5000	1
Ni	50	0.01
Pb	100	0.02
Sb	100	0.02
Se	50	0.01
Sn	100	0.02
Sr	25	0.005
Ti	50	0.01
Tl	100	0.02
V	25	0.005
Zn	100	0.02

C.5.2 CLP 2.1, CLP 4.0 CRI

In a 200-mL volumetric flask containing about 100 mL deionized water, add the following:

Add acids according to Figure 1.
0.2 mL CRI Solution 1* (SCP Science)
2.4 mL CRI Solution 2 (10 ppm Sb)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	20	.02
As	20	.02
Be	10	.01
Cd	10	.01
Co	100	.10
Cr	20	.02
Cu	50	.05
Mn	30	.03
Ni	80	.08
Pb	6	.006
Sb	10	.12
Se	10	.01
Tl	20	.02
V	100	.10
Zn	40	.04

C.5.3 CG CRI Intermediate Solution (CG CRI INT)

In a 10-mL volumetric flask containing about 8 mL deionized water, add the following:

Add acids according to Figure 1
1 mL 1000 ppm Al
0.5 mL 1000 ppm Fe

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Al	1000	100
Fe	1000	50

C.5.4 CG CRI working solution

In a 100-mL volumetric flask containing about 80 mL deionized water, add the following:

Add acids according to Figure 1
0.1 mL CRI Solution 1* (SCP Science)
1.2 mL CRI Solution 2 (10 ppm Sb)
1 mL 1000 ppm Ca
1 mL 1000 ppm K
1 mL 1000 ppm Mg
1 mL 1000 ppm Na
0.4 mL CG CRI INT

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	20	0.02
Al	1000	0.4
As	20	0.02
Ba	20	0.02
Be	10	0.01
Ca	1000	10
Cd	10	0.01
Co	100	0.1
Cr	20	0.02
Cu	50	0.05
Fe	1000	0.2
K	1000	10
Mg	1000	10
Mn	30	0.03

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Na	10	10
Ni	80	0.08
Pb	6	0.006
Sb	10	0.12
Se	10	0.01
Tl	20	0.02
V	100	0.1
Zn	40	0.04

C.5.5 CLP 5.2 CRI

In a 100-mL volumetric flask containing about 80 mL deionized water, add the following:

Add acid according to Figure 1.
1 mL CLP5.2 CRI* (High Purity)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	1	0.01
As	1.5	.015
Be	0.5	.005
Cd	0.5	.005
Co	5	.05
Cr	1	.01
Cu	2.5	.025
Mn	1.5	.015
Ni	4	.04
Pb	1	.01
Sb	6	.06
Se	3.5	.035
Tl	2.5	.025
V	5	.05
Zn	6	.06

C.5.6 Texas Risk Reduction Program (TRRP) CRI

In a 200-mL volumetric flask containing about 80 mL deionized water, add the following:

Add acid according to Figure 1.
0.1 mL CCV II A* (High Purity)
0.1 mL CCV II B* (High Purity)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	100	0.05
As	100	0.05
Ba	100	0.05
Be	100	0.05
Cd	100	0.05
Co	100	0.05
Cr	100	0.05
Cu	100	0.05
Mn	100	0.05
Mo	100	0.05
Ni	100	0.05
Pb	100	0.05
Sb	100	0.05
Se	100	0.05
Sn	100	0.05
Sr	100	0.05
Ti	100	0.05
Tl	100	0.05
V	100	0.05
Zn	100	0.05

C.6 Interference Check Solutions

C.6.1 ICP Interferent Check Solution A (ICSA)

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acid according to Figure 1.
100 mL CLPP ICS-A* (High Purity Stds.)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Al	5000	500.0
Ca	5000	500.0
Fe	2000	200.0
Mg	5000	500.0

C.6.2 ICP Interferent Check Solution AB (ICSAB)

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acid according to Figure 1.
100 mL CLPP ICS-A* (High Purity Stds.)
10 mL CLPP ICS-B4* (Inorganic Ventures)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Ag	20	0.2
Al	5000	500.0
As	10	0.1
Ba	50	0.5
Be	50	0.5
Ca	5000	500.0
Cd	100	1.0
Co	50	0.5
Cr	50	0.5
Cu	50	0.5
Fe	2000	200.0
Mg	5000	500.0
Mn	50	0.5
Ni	100	1.0
Pb	5	0.05
Sb	60	0.6
Se	5	0.05
Tl	10	0.1
V	50	0.5
Zn	100	1.0

C.7 ICP Rinse Solutions

C.7.1 ICP Rinse Solution for dilutions

In a 1000-mL volumetric flask containing about 500 mL of deionized water, add the following:

Add acids according to Figure 1.

Bring to volume with deionized water. Mix completely. Holding time is 3 months.

C.7.2 ICP Rinse Solution for instrument

Place the following in a 20-L clean container:

In about 5 L of deionized water, add the following:

1000 mL HCl
200 mL HNO_3

Bring to volume with deionized water. Mix completely. Store in a Nalgene container. Holding time is 3 months.

C.8 Arsenic (1 ppm) Profile Solution

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acids according to Figure 1, Matrix A.
1.0 mL As (1000 ppm Plasma Emission Standard) (VHG Labs, Inc.)

Bring to volume with deionized water. Solution is good for 3 months.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
As	1000	1.0

C.9 Yttrium (10 ppm)/Lithium (1000 ppm) Internal Standard

In a 20-liter carboy containing about 8000 mL deionized water, add the following:

Add acids according to Figure 1, Matrix A.
200.0 mL Yttrium (1000 mg/L Plasma Emission Standard) (VHG Labs, Inc.)
2000.0 mL Lithium (10,000 mg/L Plasma Emission Standard) (High Purity Stds.)

Add an additional 8,600 mL deionized water. Solution is good for 3 months.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Y	1000	10
Li	10000	1000

C.10 ICP Instrument Detection Limit (IDL) Stock Solution

Working solution is a 1:99 (DF100) of the following (dilute with ICP Rinse solution, Matrix "A"):

In a 100-mL volumetric flask containing about 50 mL deionized water, add the following:

1 mL HNO_3
5 mL HCl
Appropriate levels of 1000-mg/L plasma-grade standard solutions (VHG Labs, Inc.)

Bring to volume with deionized water.

Appropriate levels are determined quarterly and should be 3 to 5× the previous quarter's IDL for each analyte on each instrument. Concentrations are to be documented in the Standard Prep Log. Holding time is 3 months.

C.11 ICP Method Detection Limit (MDL) Stock Solution

In a 100-mL volumetric flask containing about 50 mL deionized water, add the following:

1 mL HNO_3
5 mL HCl
Appropriate levels of 1000-mg/L plasma-grade standard solutions (VHG Labs, Inc.)

Bring to volume with deionized water.

Appropriate levels are determined annually and should be 3 to 5× the previous year's MDL for each analyte. Concentrations are to be documented in the Standard Prep Log. The solution is then to be digested by the appropriate method. Holding time is 3 months.

C.12 Calibration Standards

C.12.1 Calibration Standard 4

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
0.1 mL 1000 ppm Al
0.1 mL 1000 ppm Ca
0.05 mL 1000 ppm Fe
0.1 mL 1000 ppm Mg

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Al	1000	1.0
Ca	1000	1.0
Fe	1000	0.5
Mg	1000	1.0

C.12.2 Calibration Standard 5

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
0.1 mL 1000 ppm K
0.2 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	1.0
Na	1000	2.0

C.12.3 Calibration Standard 6

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
0.2 mL 1000 ppm K
0.1 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	2.0
Na	1000	1.0

C.12.4 Calibration Standard 7

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
1 mL 1000 ppm K
1 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	10
Na	1000	10

C.12.5 Calibration Standard 8

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
2 mL 1000 ppm K
4.5 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	20
Na	1000	45

C.12.6 Calibration Standard 9

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
2.5 mL 1000 ppm K
4 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	25
Na	1000	40

C.12.7 Calibration Standard 10

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
3 mL 1000 ppm K
3.5 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	30
Na	1000	35

C.12.8 Calibration Standard 11

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
3.5 mL 1000 ppm K
3 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	35
Na	1000	30

C.12.9 Calibration Standard 12

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
4 mL 1000 ppm K
2.5 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	40
Na	1000	25

C.12.10 Calibration Standard 13

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
4.5 mL 1000 ppm K
2 mL 1000 ppm Na

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
K	1000	45
Na	1000	20

C.12.11 Calibration Standard 14

In a 100 mL volumetric containing about 50 mLs of deionized water, add the following:

Add acids according to Figure 1
10 mL CLPP ICS-A * (High Purity)

Bring to volume with deionized water.

Element	Initial Concentration (mg/L)	Final Concentration (mg/L)
Al	5000	500
Ca	5000	500
Fe	2000	200
Mg	5000	500

Revision Log:

Ver. #	Effective Date	Change
00	02/14/96	Previous Issue
01	02/26/99	Major changes are as follows: • Removed all references to ICAP1100 • Incorporated Procedural Amendment #2 • Added CL-CRI and NC-CRI • Added Trace Multipoint Standards • Clarified entire procedure
02	09/20/00	Major changes are as follows: • C.7. – Changed mg/L to µg/L • C.14. – Added "Holding time is 3 months" • C.15. – Added "Holding time is 3 months" • C.16. – Changed amount of diH_2O added to 8600 mL
03	06/13/03	Major changes are as follows: • Removed all references to ICAP™61 • Added LLC • Removed multi-point standards

Ver. #	Effective Date	Change
04	06/30/04	Major changes are as follows: • Added Basic Principles section • Added 5.2 CRI • C.3.3 – changed µg/L to mg/L • Updated document to Level 3 format from version 02 to version 03 • Added reagent on pg. 5
05	FEB 2 1 2005	Major changes are as follows: • All sections updated to include current vendors, current reagents, and initial and final concentration for all standards

SOPIO007C_05.DOC
012705

Prepared by: _____ Date: 1-31-05
Chemist

Approved by: _____ Date: 1.31.05
Metals Management

Approved by: _____ Date: 2/7/05
Quality Assurance

Figure 1

Matrix Matched Standards

A 1% HNO_3 5% HCl	B 10% HNO_3 4% HCl	C 6% HNO_3 4% HCl
1. 5720 CLP Waters 2. 5716 EPA 600 3. 1848 SW-846 Waters (3005A) 4. 1015 Oils 5. 2812 (3030C) 6. IDLs, Linear Ranges, 7. 6286 SW-846 Solids (Sb) 8. 4792 Dilute & Runs 9. 5281 Non-digested drinking waters	1. 5718 NPDES (conc) PWs (conc) 2. 5732 NPDES (not conc) PWs (not conc) 3. 5708 SW-846 Solids	1. 5705 TLs/EPTox (SW846-3010A) 2. 1849 CLP Solids

D. ICPMS solutions

All ICPMS solutions are to be made with ASTM Type II deionized water and Baker Analyzed acids for trace metals analysis, unless otherwise noted. All solutions will be stored at room temperature. Final volumes may be adjusted if all the components in the solution are adjusted accordingly.

Due to the sensitivity of ICPMS, all glassware and stoppers should be carefully inspected prior to use. Glassware and stoppers should also be rinsed three times with deionized water immediately prior to preparing any standards. Any standard prepared in glassware should be immediately transferred to an appropriate Nalgene container.

Most standards are prepared using 200-mL Nalgene volumetrics that have been permanently labeled with both the matrix and name of the standard that they are routinely used for. The Nalgene volumetric containing the previous lot of the same standard should always be used to prepare the new standard and should never be used to make a different standard or a different matrix. Before preparing the new standard, any standard in the volumetric should be discarded and the volumetric should be rinsed out with a few mLs of rinse of the same matrix as the standard; then proceed with instructions as per the appropriate section of this SOP.

See LOM-SOP-ES-225 for the appropriate labeling and documentation of reagent and standard preparation.

Refer to Appendix A for concentrations of stock reagents used.

D.1. ICPMS instrument rinse

In a 20-L carboy containing about 10 L deionized water, add the following:

Add acids according to Figure 1.

Bring to volume with deionized water and mix completely.

D.2. ICPMS ICB/CCB/CCS/Rinse

NOTE: The ICB/CCB/CCS/Rinse is prepared in a 20-Liter carboy for matrix D and in an 8-Liter carboy for matrix F and matrix G. It is used as the ICB, CCB and CCS check standards; as the rinse solution for dilutions; and as the common diluent for preparing other check standards of the same acid matrix. Leftover or expired ICB/CCB/CCS/Rinse may be transferred to the instrument rinse carboy of the same matrix for use as instrument rinse.

Fill the appropriate carboy approximately halfway with deionized water and add the following:

Add acids according to Figure 1.

Bring to volume with deionized water and mix completely.

Solution should be allowed to cool to room temperature before use.

Holding time is 3 months.

D.3. ICPMS calibration blank solution (S0)

In a 1000-mL volumetric flask containing about 500 mL deionized water, add the following:

Add acids according to Figure 1, using Fisher, Trace Metals Grade

Bring to volume with deionized water and mix completely.

Transfer immediately to a Nalgene bottle.

Holding time is 1 month

NOTE: The S0 may also be prepared in a 200 mL Nalgene volumetric.

D.4. ICPMS Calibration Standard 1 (S1)

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

2 mL of Standard 1 Stock (SCP Science Catalog #600-084-815).

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Holding time is 2 weeks.

Element	Final Concentration (µg/L)
Al	10000
Ca	10000
Fe	10000
K	10000
Mg	10000
Na	10000
Ag	100
As	100
Ba	100
Be	100
Cd	100
Co	100
Cr	100
Cu	100
Mn	100
Mo	100
Ni	100
Pb	100
Sb	100
Se	100
Sr	100
Ti	100
Tl	100
V	100
Zn	100

D.5. ICPMS ICV

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

1 mL of Trace CCV-I (High-Purity Standards)

0.1 mL of Trace CCV-II (High-Purity Standards)

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Holding time is 1 week.

Element	Final Concentration (µg/L)
Al	5000
Ca	5000
Fe	5000
K	5000
Mg	5000
Na	5000
Ag	50
As	50
Ba	50
Be	50
Cd	50
Co	50
Cr	50
Cu	50
Mn	50
Mo	50
Ni	50
Pb	50
Sb	50
Se	50
Sr	50
Ti	50
Tl	50
V	50
Zn	50

D.6. ICPMS CCV

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

0.5 mL TRACE CCV-I (High-Purity Standards)

0.05 mL Trace CCV-II (High-Purity Standards)

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Holding time is 1 week.

Element	Final Concentration (µg/L)
Al	2500
Ca	2500
Fe	2500
K	2500
Mg	2500
Na	2500
Ag	25
As	25
Ba	25
Be	25
Cd	25
Co	25
Cr	25
Cu	25
Mn	25
Mo	25
Ni	25
Pb	25
Sb	25
Se	25
Sr	25
Ti	25
Tl	25
V	25
Zn	25

D.7. Low Level Check Solutions

D.7.1. Low Level Check (LLC)

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

Add 0.2 mL Custom Standard 501 (VHG Labs).

Add 0.2 mL of 1.5 ppm Ni.

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Holding time is 2 weeks.

To prepare the 1.5 ppm Ni solution, dilute 0.15 mL of 1000 ppm Ni (VHG Labs) to 100 mL with matrix D rinse. Hold time for the 1.5 ppm Ni solution is 3 months.

Element	Final Concentration (µg/L)
Ag	0.5
Al	100
As	2
Ba	0.5
Be	0.1
Ca	75
Cd	0.25
Co	0.1
Cr	2
Cu	1
Fe	150
K	50
Mg	10
Mn	0.75
Mo	1
Na	200
Ni	2
Pb	1
Sb	1

Element	Final Concentration (µg/L)
Se	2
Sr	0.5
Ti	1
Tl	0.5
V	0.5
Zn	15

D.7.2. Drinking Water – Be/Tl Low Level Standard

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

Add 0.4 mL 1 ppm Tl.

Add 0.8 mL 1 ppm Be.

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Element	Final Concentration (µg/L)
Be	4
Tl	2

D.7.3. Contract Required Quantitation Limit Check (CRI)

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

Add 0.2 mL CLP-MS-CRQL (Inorganic Ventures).

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Holding time is 2 weeks.

Element	Final Concentration (µg/L)
Al	30
Ba	10
Se	5
Sb	2
Cr	2
Cu	2
Ag	1
As	1
Be	1
Cd	1
Pb	1
Ni	1
Tl	1
V	1
Zn	1
Co	0.5
Mn	0.5

D.8. ICPMS Interference check solutions

D.8.1. ICPMS ICSA solution

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

2 mL 6020 ICS-0A (Inorganic Ventures).

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Holding time is 2 weeks.

Element	Final Concentration (µg/L)
Al	10000
Ca	10000
Fe	10000
Mg	10000
K	10000
Na	10000
P	10000
S	10000
C	20000
Cl	100000
Mo	200
Ti	200

D.8.2. ICPMS ICSAB solution

Fill a 200-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

2 mL 6020 ICS-0A (Inorganic Ventures).

2 mL 6020 ICS-0B (Inorganic Ventures).

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Holding time is 2 weeks.

Element	Final Concentration (µg/L)
Al	10000
Ca	10000
Fe	10000
Mg	10000
K	10000
Na	10000
P	10000
S	10000
C	20000
Cl	100000
Mo	200
Ti	200
Ag	20
As	20
Cd	20
Co	20
Cu	20
Cr	20
Mn	20
Ni	20
Zn	20

D.9. Internal Standard Solutions

D.9.1. Internal Standard (1000 mL final volume)

Fill a 1000-mL volumetric about halfway with ICB/CCB/CCS/Rinse of the appropriate matrix, and add the following:

2 mL Internal Standard Solution 2 (VHG Labs).

0.2 mL 1000 ppm Germanium (VHG Labs).

Bring to volume with ICB/CCB/CCS/Rinse of the appropriate matrix and mix completely.

Transfer immediately to a Nalgene bottle.

Holding time is 3 months

D.9.2. Internal Standard (20-L final volume)

NOTE: For matrix D, Internal Standard is typically prepared in a 20-L carboy and transferred as needed to 1-L Nalgene bottles for daily use.

Fill a 20-L carboy with approximately 10-L of deionized water and add the following:

Add acids according to Figure 1.

Add 40 mL Internal Standard Solution 2 (VHG Labs).

Add 4 mL 1000 ppm Germanium (VHG Labs).

Bring to volume with deionized water and mix completely.

Holding time is 3 months.

Concentrations in the table below are for both Sections D.9.1 and D.9.2:

Element	Final Concentration (µg/L)
Li	500
Sc	500
Bi	100
Ga	100
In	100
Tb	100
Y	100
Ge	200

D.9.3. Internal Standard w/Methanol

NOTE: The Internal Standard w/Methanol is typically only used for the analysis of As and Se in Phillip Morris Tobacco samples (in a 6150 digest), unless otherwise directed.

In a 1000-mL Nalgene bottle add the following:

Add 900 mL of Matrix D Internal Standard.

Add 100 mL methanol (Sigma-Aldrich).

Mix completely.

Holding time is 3 months.

Element	Final Concentration (µg/L)
Li	450
Sc	450
Bi	90
Ga	90
In	90
Tb	90
Y	90
Ge	180

D.10. Tuning solution

This solution is a pre-mix that is spiked with 0.1 mL of 1000 ppm Li (per Liter of Perkin Elmer Pure, Elan 6100 Setup/Stab/Masscal Solution). This solution is used for Daily Tuning and Daily Performance checks as well as for various instrument optimization procedures.

Element	Final Concentration (µg/L)
Li	100
Mg	10
Cu	10
Rh	10
Cd	10
In	10
Ba	10
Ce	10
Pb	10
U	10

Solution expires according to manufacturer's expiration date on bottle.

NOTE: The purchased tuning solution contains copper, barium and cadmium. These elements are not evaluated during the instrument tuning procedure.

D.11. Dual Detector Calibration Solution

Fill a 1000-mL volumetric about halfway with matrix D ICB/CCB/CCS/Rinse, and add the following:

10 mL Internal Standard Solution 2 (VHG Labs)

0.2 mL 1000 ppm Germanium (VHG Labs)

0.2 mL Trace CCV-I (High Purity Standards)

2 mL Trace CCV-II (High Purity Standards)

Bring to volume with matrix D ICB/CCB/CCS/Rinse and mix completely.

Holding time is 3 months

Element	Final Concentration (µg/L)
Li	500
Sc	500
Y	100
In	100
Tb	100
Ga	100
Bi	100
Ge	200
Ag	200
Al	200
As	200
Ba	200
Be	200
Ca	200
Cd	200
Co	200
Cr	200

Element	Final Concentration (µg/L)
Cu	200
Fe	200
K	200
Mg	200
Mn	200
Mo	200
Na	200
Ni	200
Pb	200
Sb	200
Se	200
Sr	200
Ti	200
Tl	200
V	200
Zn	200

D.12. Auto Lens Calibration Solution

Fill a 1000-mL volumetric about halfway with matrix D ICB/CCB/CCS/Rinse, and add the following:

0.2 mL Internal Standard Solution 2 (VHG Labs).

1 mL 10 ppm Be & Co solution (prepared by adding 0.1 mL of 1000 ppm Be and 0.1 mL 1000 ppm Co into a 10-mL volumetric flask and bringing to volume with matrix D ICB/CCB/CCS/Rinse).

Bring to volume with matrix D ICB/CCB/CCS/Rinse and mix completely.

Holding time is 3 months

Element	Final Concentration (µg/L)
Li	50
Sc	50
Bi	10
Ga	10
In	10
Tb	10
Y	10
Be	10
Co	10

D.13. Non-CLP PDS Spike

Fill a 100-mL volumetric about halfway with matrix D ICB/CCB/CCS/Rinse, and add the following:

Add 10 mL Custom Standard 501 (VHG Labs).

Add 10 mL 1.5 ppm Ni (see Section D.7.1.).

Bring to volume with matrix D ICB/CCB/CCS/Rinse and mix completely.

Holding time is 3 months.

Element	Final Concentration (µg/L)	Concentration in Sample (µg/L)
Ag	50	1
Al	10000	200
As	200	4
Ba	50	1
Be	10	0.2
Ca	7500	150
Cd	25	0.5
Co	10	0.2

Element	Final Concentration (µg/L)	Concentration in Sample (µg/L)
Cr	200	4
Cu	100	2
Fe	15000	300
K	5000	100
Mg	1000	20
Mn	75	1.5
Mo	100	2
Na	20000	400
Ni	200	4
Pb	100	2
Sb	100	2
Se	200	4
Sr	50	1
Ti	100	2
Tl	50	1
V	50	1
Zn	1500	30

The spiked sample (PDS) is prepared by pipetting 0.1 mL of the Non-CLP PDS Spike into a 5-mL VF and bringing to volume with the background sample (U).

D.14. ICPMS LRS solution

Fill a 200-mL volumetric about halfway with matrix D ICB/CCB/CCS/Rinse, and add the following:

2 mL TRACE CCV-I (High-Purity Standards)

1 mL Trace CCV-II (High-Purity Standards)

Bring to volume with Matrix D ICB/CCB/CCS/Rinse and mix completely.

Holding time is 1 week.

Element	Final Concentration (µg/L)
Al	10000
Ca	10000
Fe	10000
K	10000
Mg	10000
Na	10000
Ag	500
As	500
Ba	500
Be	500
Cd	500
Co	500
Cr	500
Cu	500
Mn	500
Mo	500
Ni	500
Pb	500
Sb	500
Se	500
Sr	500
Ti	500
Tl	500
V	500
Zn	500

Revision Log:

Ver. #	Effective Date	Change
00	09/15/93	Previous issue
01	05/30/00	Major changes are as follows: • Deleted section – no longer in use.
02	06/26/03	Major changes are as follows: • Reformatted to Level 3 • Added ICPMS standards and solutions
03	12/19/03	Major changes are as follows: • Updated ICPMS standards section • Changed instrument rinse to reflect new acid concentration • Changed S1, S2, S3, ICV, CCV, ICSA, ICSAB and Internal Standard solutions to reflect new concentrations • Added ICB/CCB, LLC, Dual Detector Calibration, and Auto Lens Calibration Solutions • Updated Tuning Solution information • Updated Figure 1
04	03/25/04	Major changes are as follows: • Changed hold time of solutions
05	10/08/04	Major changes are as follows: • Updated sections D.1.2 and D.9 to reflect the practice of matrix matching the instrument rinse and internal standard to the matrix of samples currently being analyzed. • Updated sections D.5 and D.7 to reflect changes in the preparation of the CCV and LLC check standards. • Added section D.13 for Non-CLP PDS Spike.

Ver. #	Effective Date	Change
06	11/17/04	Major changes are as follows: • Solution and reagent volumes were changed to reflect the volumetric flask size most commonly used. • Added changes in S1, S2 and S3 to reflect changes to the calibration for Na, Mg, Al, K, Ca and Fe. • Added changes to ICV, CCV, LLC, Internal Standard, Dual Detector Calibration, Auto Lens Calibration, and NonPDS Spike solutions to reflect concentration changes and changes to the stock reagents used. • Minor clarifications added throughout the procedure. • Added Appendix A which contains a list of the stock reagents used as well as the concentrations of the elements they contain. • Added Section D.14 for CRI check standard.
07	11/18/04	Major changes are as follows: • Updated section D.7.1
08	11/03/05	Major changes are as follows: • Section D.10 – updated this section to indicate which elements are not evaluated in the Tuning Solution
09	11/30/05	Major changes are as follows: • Removed references to S2, S3 and all references to Sn • Updated concentrations and instructions for LLC, PDS, S1, AutoLens, ICV and CCV solutions and changed all concentrations to µg/L. • Modified instructions to include use of matrix matched ICB/CCB/CCS/Rinse as a common diluent for preparing standards • Updated Figure 1 and Appendix A

Ver. #	Effective Date	Change
10	JUL 07 2006	Major changes are as follows: • Updated Section D.2 to reflect the actual volume for the carboys used for matrix F and matrix G. • Updated Sections D.7.1 and D.13 to reflect addition of 1.5 ppm Ni solution to LLC and PDS. • Updated Section D.9 to include instructions for preparing Internal Standard w/Methanol. • Updated Figure 1 and Appendix A. • Minor clarifications throughout the procedure.

SOPIO007D_10.DOC
061306

Prepared by: David K Beck — Date: 6-15-06
Chemist

Approved by: [signature] — Date: 6/21/06
Metals Management

Approved by: Elaine Stoltzfus — Date: 6/23/06
Quality Assurance

Figure 1

Matrix Matched Standards

Final Volume ↓	D 6% HNO_3	E 2% HNO_3	F 2% HNO_3 1% HCl	G 1% HNO_3
1000 mL	60 mL HNO_3	20 mL HNO_3	20 mL HNO_3 10 mL HCl	10 mL HNO_3
8 L	480 mL HNO_3	160 mL HNO_3	160 mL HNO_3 80 mL HCl	80 mL HNO_3
20 L	1200 mL HNO_3	400 mL HNO_3	400 mL HNO_3 200 mL HCl	200 mL HNO_3
	6020	200.8 Soils	CLP 5.2 > 1.0 NTU 200.8 >1.0 NTU	CLP 5.2 < 1.0 NTU 200.8 < 1.0 NTU

Appendix A

This Appendix contains a list of the stock reagents referenced in this procedure as well as the approximate concentrations. For actual concentrations see the appropriate Certificate of Analysis kept on file in the Metals Department.

Acids and Reagents

Nitric Acid, Trace Metal Grade (Fisher Scientific)
Hydrochloric Acid, Trace Metal Grade (Fisher Scientific)
Nitric Acid, ACS (J.T. Baker)
Hydrochloric Acid, ACS (J.T. Baker)
Methanol (Sigma-Aldrich)

Multielement Stock Standards

Standard 1 Stock (SCP Science, Catalog #600-084-815): Al, Ca, Fe, K, Mg and Na @ 1000 ug/mL; Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Sr, Ti, Tl, V and Zn @ 10 ug/mL.

Trace CCV-I (High-Purity Standards): Al, Ca, Fe, K, Mg and Na @ 1000 ug/mL.

Trace CCV-2 (High-Purity Standards): Sb, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, As, Sr, Tl, Sn, Ti, V and Zn @ 100 ug/mL.

Custom Standard 501 (VHG Labs): Be and Co @ 0.1 ug/mL; Cd @ 0.25 ug/mL; Ba, Ni, Ag, Sr, Tl and V @ 0.5 ug/mL; Mn @ 0.75 ug/mL; Sb, Cu, Pb, Mo, Sn and Ti @ 1 ug/mL; As, Cr and Se @ 2 ug/mL; Mg @ 10 ug/mL; Zn @ 15 ug/mL; K @ 50 ug/mL; Ca @ 75 ug/mL; Al @ 100 ug/mL; Fe @ 150 ug/mL; Na @ 200 ug/mL.

6020 ICS-0A (Inorganic Ventures): Cl @ 10,000 ug/mL; C @ 2,000 ug/mL; Al, Ca, Fe, Mg, P, K, Na and S @ 1,000 ug/mL; Mo and Ti @ 20 ug/mL.

6020 ICS-0B (Inorganic Ventures): As, Cd, Cr, Co, Cu, Mn, Ni, Ag and Zn @ 2 ug/mL.

Internal Standard Solution 2 (VHG Labs): Li and Sc @ 250 ug/mL; Bi, Ga, In, Tb and Y @ 50 ug/mL.

CLP-MS-CRQL (Inorganic Ventures): Al @ 30 ug/mL; Ba @ 10 ug/mL; Se @ 5 ug/mL; Sb, Cr and Cu @ 2 ug/mL; Ag, As, Be, Cd, Pb, Ni, Tl, V and Zn @ 1 ug/mL; Co and Mn @ 0.5 ug/mL.

Procedural Amendment #1

Procedure Title: ICPMS Solutions

Reasons for addition(s) or change(s): Change in stock used for LLC

Samples or project affected: All

List change(s) or addition(s) (specify which section):

In Section D.7.1. Low Level Check (LLC) *– The LLC is now prepared with 0.2 mL of Custom Standard 601 diluted to 200 mL with ICB/CCB/CCS/Rinse of the appropriate matrix. Custom Standard 501 and the 1.5 ppm Ni are no longer used. The final concentrations of the LLC are as follows: Be @ 0.2 µg/L; Cd @ 0.25 µg/L; Ag, Ba & Tl @ 0.5 µg/L; Cu, Pb & Sb @ 1.0 µg/L; As, Cr, Ni & Se @ 2.0 µg/L; Zn @ 15 µg/L.*

In Section D.13. Non-CLP PDS Spike *– The Non-CLP PDS Spike is now prepared with 10 mL of Custom Standard 601 diluted to 100 mL with Matrix D ICB/CCB/CCS/Rinse. Custom Standard 501 and the 1.5 ppm Ni are no longer used. The final concentrations of the Non-CLP PDS Spike are as follows: Be @ 20 µg/L; Cd @ 25 µg/L; Ag, Ba & Tl @ 50 µg/L; Cu, Pb & Sb @ 100 µg/L; As, Cr, Ni & Se @ 200 µg/L; Zn @ 1500 µg/L. The final concentrations of the PDS Sample (0.1 mL of Non-CLP PDS Spike to 5 mL with sample) are as follows: Be @ 0.4 µg/L; Cd @ 0.5 µg/L; Ag, Ba & Tl @ 1 µg/L; Cu, Pb & Sb @ 2 µg/L; As, Cr, Ni & Se @ 4 µg/L; Zn @ 30 µg/L.*

In Appendix A *the reference to Custom Standard 501 is deleted and replaced with "Custom Standard 601 (VHG Labs): Be @ 0.2 µg/mL; Cd @ 0.25 µg/mL; Ag, Ba & Tl @ 0.5 µg/mL; Cu, Pb & Sb @ 1.0 µg/mL; As, Cr, Ni & Se @ 2.0 µg/mL; Zn @ 15 µg/mL."*

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Prepared by: David A Beck Date: 9-14-06
Chemist

Approved by: [signature] Date: 9.15.06
Metals Management

Approved by: Dorothy McClave Date: 9/18/06
Quality Assurance

E. Mercury solutions

E.1 Intermediate calibration standards

E.1.1 Hg intermediate standards (10 mg/L) - Prepare a 10 mg/L Hg solution from 1000 mg/L Hg standard solution and identify it as "CONTROL" (Calibration Verification). Prepare a second 10 mg/L Hg solution from a different 1000 mg/L standard source and identify it as "CAL" (Calibration). Prepare each solution as described in the following paragraph.

Pipette 1.0 mL of 1000 mg/L Hg standard solution into a 100-mL volumetric flask. Add 25 mL water, 1 mL HNO_3, and 0.01 g of solid potassium dichromate ($K_2Cr_2O_7$). Swirl to dissolve the dichromate. Dilute to volume with water. Store in a polyethylene bottle. Identify as indicated above. The holding time for this solution is 6 months.

E.1.2 Hg intermediate standard (1.0 mg/L) - Prepare one from the CAL and one from the CONTROL 10 mg/L intermediate standards. Pipette 1.0 mL of 10 mg/L Hg into a 10-mL volumetric flask. Dilute to volume with 0.15% HNO_3. Mark CAL and CONTROL, respectively. Prepare daily.

E.1.3 Hg Intermediate (0.1 mg/L) - Prepare one from the CAL and one from the CONTROL 1.0 mg/L intermediate standards. Pipette 1.0 mL of 1.0 mg/L Hg into a 10-mL volumetric flask. Dilute to volume with 0.15% HNO_3. Mark CAL and CONTROL, respectively. Prepare daily.

E.2 Manual water digestion standards

NOTE: If a water bath is used, the samples/standards are digested in 250-mL Erlenmeyer flasks. If a block digester is used, the samples are digested in polypropylene containers.

E.2.1 Hg calibration standards manual digestion - Prepare the calibration standards daily by pipetting the following CAL Intermediate Standard volumes into 100 mL of water into the appropriate sample/standard container:

Quantity	Calibration Standard Concentration (mg/L)	Volume of Intermediate Standard Added (mL)	Intermediate Standard Concentration (mg/L)
2	Blank	---	---
1	.0002	0.2	0.1
1	.0005	0.5	0.1
2	.0010	0.1	1.0
1	.0025	0.25	1.0
1	.0050	0.5	1.0

Digest the standards using digestion Method 821. See Section E.2.2 of this SOP or see the digestion method for details.

E.2.2 Hg initial calibration verification (ICV) standard (0.0025 mg/L) - Pour 100 mL of water into the appropriate sample/standard container. Pipette 0.25 mL of CONTROL Intermediate Standard (1.0 mg/L Hg) into the container. Add 2.5 mL HNO_3, 5 mL H_2SO_4, 15 mL $KMnO_4$ (5%), and allow to stand for 15 minutes. Add 8 mL $K_2S_2O_8$ (5%) and place in a block digestor or water bath, depending on digestion procedure, for 2 hours. Cool. Prior to analysis, add 6 mL sodium chloride/hydroxylamine hydrochloride solution. Transfer to a 100-mL volumetric flask, dilute to volume with water, and reserve for analysis. Prepare daily.

E.2.3 Hg initial calibration verification (ICV) standard (0.0020 mg/L) for CLP ILM05.2 only- Pour 100 mL of water into the appropriate sample/standard container. Pipette 0.20 mL of CONTROL Intermediate Standard (1.0 mg/L Hg) into the container. Add 2.5 mL HNO_3, 5 mL H_2SO_4, 5 mL $KMnO_4$ (5%), and allow to stand for 15 minutes. Add 8 mL $K_2S_2O_8$ (5%) and place in a block digestor or water bath, depending on digestion procedure, for 2 hours. Cool. Prior to analysis, add 6 mL sodium chloride/hydroxylamine hydrochloride solution. Transfer to a 100-mL volumetric flask, dilute to volume with water, and reserve for analysis. Prepare daily.

E.2.4 Hg low-level check (CRA) standard (0.0002 mg/L) - Pour 100 mL of water into the appropriate sample/standard container. Pipette 0.2 mL of CONTROL Intermediate Standard (0.1 mg/L Hg) into the container. Continue as described in section E.2.2.

E.2.5 Hg continuing calibration verification (CCV) standard – Pour approximately 100 mL of water into the appropriate sample/standard container. Pipette 0.10 mL of control intermediate standard (1.0 mg/L Hg) into the container. Continue as described in Section E.2.2.

E.3 Leeman Labs AP200II digestion standards

E.3.1 0.15% Nitric acid solution (0.15% HNO_3) – Dilute 1.5 mL of concentrated nitric acid to 1000 mL with deionized water. This solution is good for 6 months. Store at room temperature.

E.3.2 Hg calibration standards for the Leeman Labs AP200II preparation system – Prepare the calibration standards daily by pipetting the following CAL intermediate standard volume into a 100-mL volumetric flask. Dilute to volume with deionized water.

Quantity	Calibration Standard Concentration (mg/L)	Volume of Intermediate Standard Added (mL)	Intermediate Standard Concentration (mg/L)
1	Blank	---	---
1	.0002	0.2	0.1
1	.0005	0.5	0.1
1	.0010	0.1	1.0
1	.0025	0.25	1.0
1	.0050	0.5	1.0

Digest the standards using digestion Method 821 with the Leeman Labs AP200II.

E.3.3 Hg initial calibration verification (ICV) standard (0.0025 mg/L) – Pour approximately 50 mL of 0.15% HNO_3 into a 100-mL volumetric flask. Pipette 0.25 mL of control intermediate standard (1.0 mg/L Hg) into the flask and dilute to volume with deionized water.

E.3.4 Hg initial calibration verification (ICV) standard (0.0020 mg/L) for CLP ILM05.2 only – Pour approximately 50 mL of 0.15% HNO_3 into a 100-mL volumetric flask. Pipette 0.20 mL of control intermediate standard (1.0 mg/L Hg) into the flask and dilute to volume with deionized water.

E.3.5 Hg continuing calibration verification (CCV) standard and laboratory control sample (LCS) (0.0010 mg/L) – Pour approximately 50 mL of 0.15% HNO_3 into a 100-mL volumetric flask. Pipette 0.10 mL of control intermediate standard (1.0 mg/L Hg) into the flask and dilute to volume with deionized water.

E.3.6 Hg low-level check (CRA) standard (0.0002 mg/L) – Pour approximately 50 mL of 0.15% HNO_3 into a 100-mL volumetric flask. Pipette 0.20 mL of control intermediate standard (0.1 mg/L Hg) into the flask and dilute to volume with deionized water.

E.4 General solutions

E.4.1 Potassium permanganate solution (5%) - Weigh approximately 50 g of potassium permanganate ($KMnO_4$) into a 600-mL beaker. Transfer the $KMnO_4$ into a 1000-mL volumetric flask using water. Dilute to approximately 950 mL with water. Using a stir plate, stir until the $KMnO_4$ is dissolved. Remove the spin bar and dilute to volume with water.

E.4.2 Potassium persulfate solution (5%) - Weigh approximately 25 g of potassium persulfate ($K_2S_2O_8$) into a small beaker. Transfer the $K_2S_2O_8$ into a 500-mL volumetric flask using water. Add approximately 250 mL water. Swirl to dissolve the $K_2S_2O_8$. (Gentle heat may be necessary.) Dilute to volume with water.

E.4.3 Sodium chloride/hydroxylamine hydrochloride solution - Weigh approximately 120 g of sodium chloride (NaCl) into a 400-mL beaker. Transfer, using water, to a 1000-mL volumetric flask. Weigh approximately 120 g of hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) into a 400-mL beaker. Transfer, using water, to the 1000-mL volumetric flask containing the NaCl. Add water and swirl to dissolve solids and dilute to volume with water.

E.4.5 Stannous chloride solution (10%) Leeman Labs PS200II – Weigh approximately 100 g of stannous chloride ($SnCl_2$) into a 250-mL beaker. Transfer using water to a 1000-mL volumetric flask. Add 100 mL of HCl and dilute to volume with deionized water.

E.4.6 0.15% nitric acid solution (0.15% HNO_3) – Dilute 1.5 mL of concentrated nitric acid to 1000-mL with deionized water. This solution is good for 6 months. Store at room temperature.

NOTE: As long as the correct rations are maintained, solutions may be prepared using multiples of indicated weights and volumes.

Revision Log:

Ver. #	Effective Date	Change
00	10/16/95	Previous Issue
01	05/08/97	Major changes are as follows: • Section E.3 - Use of 0.15% HNO_3 instead of 1% HNO_3 • Section E.4.6 - Preparation of 0.15% HNO_3 • Changes made as per methods 7470 SW-846 and 245.1 EPA 600
02	07/06/99	Major changes are as follows: • Incorporate Procedural Amendment #1 • Section E.1.1 - Remove "SPEX" and "or equivalent" • Section E.4.4 - Delete • Change all references of AP200 to AP200II
03	08/07/00	Major changes are as follows: • E. 2.2., E.2.3., E.2.4. – Changed 250 mL Erlenmeyer flasks to polypropylene sample containers, changed 95°C water bath to hot block, Removed part of sentence "or until solution volume is 90 mL or less." • E2.2. & E.3.3. – Changed ICV concentration from 0.0020 mg/L to 0.0025 mg/L, changed pipette amount from 0.2 mL to 0.25 mL.
04	06/28/01	Major changes are as follows: • Clarified SOP throughout regarding use of polypropylene containers Vs. Erlenmeyer flasks for manual digestion
05	07/18/01	Major changes are as follows: • E.2. – Clarified sample container • E.2. – Changed Note

Ver. #	Effective Date	Change
06	12/15/04	Major changes are as follows: • Insert ICV (0.0020 mg/L) to sections E.2 and E.3 • E.1.2 – Changed dilute to volume with water to dilute to volume with 0.15% HNO_3 • E.3.1 and E.4.5 – Solution good for 1 month changed to 6 months • E.3.2 – Changed dilute to volume with 0.15% HNO3 to dilute to volume with water
07	MAR 3 1 2006	Major changes are as follows: • E1.3 – Changed dilute to volume with water to dilute to volume with 0.15% HNO3 • E3.2 to E3.6 – Changed dilute to volume with 0.15% HNO3 to dilute to volume with water • E2.2 & E2.3 – Changed volume of KMnO4 from 5 mL to 15 mL • E2.2 – Changed volume of sodium chloride/hydroxylamine hydrochloride solution from 2 mL to 6 mL • Added Note

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031406

Prepared by: Jennifer Mayer Date: 03/14/06
Specialist, Group Leader

Approved by: Date: 3.15.06
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Approved by: Elaine Stoltzfus Date: 3/17/06
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SOP-IO-014.18
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Quality Control Procedures for ICP

Approvals:

Prepared by: _____ Date: 9/13/06
Senior Specialist

Approved by: _____ Date: 9.13.06
Metals Management

Approved by: Ann Lemmo Date: 9/13/06
Quality Assurance

Revision Log:

Ver. #	Effective Date	Change
00	09/04/96	Previous Issue

01	01/30/98	Major changes are as follows:
		• References added for SW-846 6010B and EPA 600 200.7/R-94-111
		• Added definitions for SIC and LRL solutions
		• The following throughout the Procedure section:
		  • Added 6010B criteria for ICB, CCV, and CCB
		  • CRI and LRL check added
		  • Modified EPA 600 criteria for the LCS
		  • Added EPA 600 criteria for MS, PDS, and SD
		  • Added GLP "real-time" printout requirement
		  • Applied changes to Table I

02	02/04/99	Major changes are as follows: • Added Personnel Training and Qualifications section. • Removed references to LRL and SIC solutions which are no longer used (sections B.8, B.9 in Definitions section, and B.5, B.6 in Procedure section). • Added internal standard check (C.7). • Added 6010B rule for evaluating samples is CCB is >3× IDL (B.4.b. (2)).
03	10/21/99	Major changes are as follows: • Incorporated Procedural Amendment #1. • Added EPA 600 (PW) updates to ICV (B.3.a.) and PDS (B.9.b.). • Added GLP real-time run requirement (D.5.).
04	04/06/00	Major changes are as follows: • Added reference to ILM04.1 • Added linear range requirement for EPA 600 • Amended carryover criteria • Amended CRI acceptance criteria

Ver. #	Effective Date	Change
05	05/17/01	Major changes are as follows: • Added Cross Reference section • Referred to statistical windows for LCS/LCSD and MS/MSD calculated % recoveries • Removed all references to ICAP 61 • Added where analysts can find current LOQs • Added reference for spike added
06	03/17/03	Major changes are as follows: • Changed wording for clarification throughout and added requirements for CLP 5.2 • Procedure – B.6. Added LLC criteria • Procedure – D. 4. Changed WANG to Parallax • Updated procedure due to change in internal documentation system.
07	03/20/03	Major changes are as follows: • Changed Title • Updated entire procedure • Added Tables II, III, and IV
08	07/28/03	Major changes are as follows: • Updated Definitions section • Added Wavelengths to Routine Methods section • Clarified Procedure section • Updated Tables I, II, III, and IV
09	10/16/03	Major changes are as follows: • Removed regular analysis numbers from Routine Methods section • Clarified Procedure section
10	03/05/04	Major changes are as follows: • Added Basic Principle • Added in Procedure #13 and a NOTE
11	02/10/05	Major changes are as follows: • Updated Definitions section • Updated Table 3

Ver. #	Effective Date	Change
12	04/06/05	Major changes are as follows: • Added ICV2 requirements for PW, EW, and U4 samples. • Updated software error code information in Appendix I.
13	05/03/05	Major changes are as follows: • Procedure section A.12 and 14, removed U4 reference • Tables at the end of the procedure, added statistical acceptance for LLC
14	07/15/05	Major changes are as follows: • Added review and verification information to sections A, B, C.
15	11/10/05	Major changes are as follows: • Procedure A.7 • Removed Table I • Deleted references to High Standards • Deleted reference to error coding runs.
16	02/14/06	Major changes are as follows: • Updated Cross Reference and Procedure sections
17	09/07/06	Major changes are as follows: • Moved signature page and revision log to front of document to reflect current procedures • Procedure A.15. – added note about the internal standard • Updated the tables I, II and III • Updated Appendix 1.B • Updated/clarified Note in section A.15
18	SEP 27 2006	Major changes are as follows: • Updated (and clarified) **Note** in section A.15.

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091206

Reference:

1. ILM02.1, Section E., USEPA CLP Statement of Work, March 1990.
2. ILM04.0, Section E., USEPA CLP Statement of Work.
3. ILM05.2, Exhibit D/ICP-AES, USEPA CLP Statement of Work.
4. Method 6010A, USEPA SW-846, 7/92.
5. Method 6010B, USEPA SW-846, 12/96.
6. Method 200.7, USEPA 600/4-91/010.
7. Method 200.7, USEPA 600/R-94-111.

Cross Reference:

Document	Document Title
LOM-SOP-ES-207	Establishing Control Limits
LOM-SOP-ES-222	Instrument and Equipment Maintenance and Calibration
SOP-IO-007	Preparation of Standards and Solutions
SOP-IO-012	Calculations Used by the Inorganics Group

Purpose:

This SOP is designed to provide consistent guidelines for the evaluation of ICP data.

Scope:

This procedure applies to analyses performed in Environmental Sciences using ICP for identification and quantitation of metallic constituents.

Definitions:

Batch and instrument QC

1. Analytical Batch – A group of field samples that are digested and analyzed together. A batch consists of no more than 10 samples for EPA 600 methods or no more than 20 samples for other methods.

2. Analytical Samples – Analytical sample is defined as any solution introduced into an instrument on which an analysis is performed, excluding instrument calibration, ICV, ICB, CCV, CCB, and tunes. Analytical samples include undiluted and diluted samples, matrix spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, ICSs, CRIs, LLCs, PBs, LCSs, PEs, and Linear Range Samples (LRSs).

3. Background Sample (U) – The original sample from which the batch QC is derived. The background sample is either site specific or randomly selected.

4. Continuing Calibration Blank (CCB) – A reagent blank run immediately after every CCV. This is used to monitor the stability of the low end of the calibration.

5. Continuing Calibration Verification (CCV) – A mid-range standard run at a frequency of 10% (every ten samples) throughout the run. This is used to monitor instrument drift.

6. Contract Required Detection Limit (CRI) – A standard analyzed at the Contract Laboratory Program (CLP) required detection limit. This sample verifies linearity near the limit of quantitation.

7. Duplicate Sample (D) – A replicate of the original sample, processed in parallel. This sample is used to provide a measure of the in-lab repeatability (precision) of the analytical process. The duplicate sample is either site specific or randomly selected.

8. Initial Calibration Verification (ICV) – This is a standard near the middle of the calibration range, prepared from a different source than the calibration standards. It is used to prove that the instrument is calibrated correctly at the start of the run.

9. Initial Calibration Blank (ICB) – This is a standard reagent blank, used to prove that the low end of the calibration is acceptable. It must be run immediately after the ICV.

10. Interelement Correction Standard-A (ICSA) – A standard containing high concentrations of commonly interfering elements. It is used to assess the spectral interferences due to matrix elements that can normally be expected to be found in a sample.

11. Interelement Correction Standard-AB (ICSAB) – A standard containing both interfering elements and target analytes, run immediately after the ICSA. It is used to demonstrate the effectiveness of the interelement correction factors in use.

12. Instrument detection limit (IDL) – A value determined from analyzing 7 standard solutions (undigested) at a concentration 3× to 5× the anticipated IDL on three nonconsecutive days. The standard deviation obtained for these multiplied by 3 is the IDL. These must be performed quarterly on each instrument used for an analyte.

    For CLP 5.2, an NP1MDL is determined by analyzing 7 non-digested standards at a concentration of 3× to 5× the expected IDL. These are performed quarterly on all instruments used for an analyte.

13. Laboratory Control Sample (LCS) – A spike reagent blank of known composition carried through the digestion process. It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes.

14. Laboratory Control Sample Duplicate (LCSD) – This is a duplicate of the matrix-matched synthetic sample of known composition. It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes. It is also used as a measure of the precision of the analytical process.

15. Limit of Quantitation (LOQ) – The level above which quantitative results may be obtained with a specified degree of confidence. It is based on a value 3× to 5× the MDL. CLP 4.0 samples are reported using the IDL and CRDL, CLP 5.2 samples are reported using the MDL and CRQL; the statement of work for the programs specifies required limits.

16. Low Level Check Standard (LLC) – A low-level standard used to monitor the performance of the instrument near the detection limit.

17. Matrix Spike Duplicate (MSD) – A duplicate of the Matrix Spike Sample (R) which is a replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure. It is also used as a measure of the precision of the analytical process. The matrix spike duplicate sample is either site specific or randomly selected.

18. Matrix Spike Sample (R) – A replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure. The matrix spike sample is either site specific or randomly selected.

19. Method Detection Limit (MDL) – The minimum concentration of a substance that can be reported with 99% confidence that the analyte concentration is greater than 0. It is determined by analyzing 7 digested standards at an estimated concentration 2.5× to 5× the signal/noise ratio. MDLs are performed on all instruments used to determine each analyte. MDLs are not listed in this SOP. They can be found in the LIMS system due to the fact that they change on a regular basis.

20. Post Digestion Spike (PDS) – This sample is a spike of the Background Sample prepared after digestion, at the time of analysis. It is used to determine if out-of-specification spike recoveries are due to problems in the digestion or are matrix related.

21. Preparation Blank (PB) – This is a reagent blank carried through the entire digestion procedure. It is used to determine if contamination has occurred during the digestion procedure.

22. Serial Dilution (SD) – This sample is a 1:4 (5×) dilution of the Background Sample, prepared after the digestion. It is used to indicate the presence of any matrix effects that could cause a nonlinear response at the instrument.

23. Linear Range (LR) – This is the highest sample concentration that can be read at ±5% of true value.

Basic Principles:

The Quality Control requirements for accurately reviewing and verifying a sample data run are listed in this SOP.

Personnel Training and Qualifications:

1. Review and understanding of this procedure
2. Trainee observing trained analyst performing the procedure
3. Trainer observing trainee performing the procedure
4. Review of the trainee's data by trainer
5. Documentation of critical steps in the training process
6. Demonstration of proficiency by being able to independently review ICP data
7. Analysts and verifiers must have read all applicable internal SOPs including this SOP

Routine Methods:

Elements routinely analyzed on the Thermo Jarrell Ash ICAP™ 61E Trace Spectrometer include Lancaster Laboratories methods:

Element	Waters Analysis #	Solids Analysis #	Wavelength (nm)
As	7035	6935	189.04
Se	7036	6936	196.02
B	8014	7914	249.67
Tl	7022	6925	190.86
Al	1743	1643	308.21
Sb	7044	6944	206.83
Ba	7046	6946	493.40
Be	7047	6947	313.04
Cd	7049	6949	226.50
Ca	1750	1650	317.93
Cr	7051	6951	267.71
Co	7052	6952	228.61
Cu	7053	6953	324.75
Fe	1754	1654	271.44 or 259.94
Pb	7055	6955	220.35
Mg	1757	1657	279.07
Mn	7058	6958	257.61
Mo	7060	6960	202.03
Ni	7061	6961	231.60
K	1762	1662	766.49
Ag	7066	6966	328.06
Na	1767	1667	330.23
Sr	8068	7968	421.55
Sn	7069	6969	189.98
Ti	7070	6970	334.94
V	7071	6971	292.40
Zn	7072	6972	213.85 or 206.20

Procedure:

A. Raw data quality checks

1. Make sure that the run is correctly labeled, dated, and signed and that the corresponding cover sheet is attached to the front of the run. Also check that the print is dark enough for data packages to easily copy.

2. Check to see that the autosampler table printout is with the run and has a review signature from the analyst and run importer.

3. Check that the sample numbers, weights, and volumes on the run cover sheet correctly match the batch sheet. Review the batch sheet for special comments and due dates.

4. Check that the run protocol has been selected correctly for specific client requirements. Also review the sample's state for additional requirements. North Carolina samples must be diluted to < the highest standard, and some Kentucky samples use Method 6010A, Update II.

5. For calculations used by the inorganics groups see SOP-IO-012.

6. For run and batch QC frequency, acceptance criteria and corrective action refer to Tables I, II, and III. For information on statistical windows refer to LOM-SOP-ES-207.

7. For spike levels of run QC see SOP-IO-007, Section C.

8. For spike levels of batch QC see SOP-IO-007, Sections G and H.

9. LOQs are available to analysts in the LIMS and on charts that are updated as needed.

10. Check to make sure that all results are below the linear range limit. If a sample reading is above the linear range, then reread the sample at an appropriate dilution. For EPA 600, reread the sample at a dilution if it reads >90% of the linear range. For CLP 5.2, the diluted sample reading must fall within the upper half of the linear range. Verifiers will footnote the coversheet that all dilutions were performed correctly by comparing to the previous undiluted sample data.

11. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings <(-LOQ). Comments will be added during verification to any non-detect sample readings that were diluted due to <(-LOQ).

12. Check for carryover between samples. Sample RSD >20%, with a concentration > the LOQ decreasing progressively over time (i.e., Reading 3<2<1). Flag any suspect samples for reread.

13. For TCLP samples, an MSA (method of standard additions) is required if the sample concentration falls between 80% to 100% of the regulatory limits.

14. For all EW samples (samples from public drinking water sources), check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once so that the supplier can be notified. Suppliers must be notified within 1 hour.

Analyte	MCL (mg/L)
Sb	0.006
As	0.05
Ba	2 (1)**
Be	0.004
Cd	0.005
Cr	0.1 (0.05)**
Se	0.05 (0.01)**
Tl	0.002
Al*	0.2
Cu*	1.0
Fe*	0.3
Mn*	0.05
Ag*	0.1 (0.05)**
Zn*	5.0

*Secondary regulated contaminants

**The federal MCLs for these analytes are greater than Pennsylvania MCLs. The numbers in parentheses are the MCLs effective in Pennsylvania

15. Check the internal standard (yttrium) level for the entire run. If the yttrium reading for any sample is >130% of the reading for S0, then reread the sample at a dilution.

NOTE: The internal standard is added in equal concentration to all of the samples and standards. It is added to the sample via a dedicated line on the peristaltic pump. The analytical lines referenced to an internal standard report a corrected concentration value based on the ratio of analyte to internal standard intensities. All of the calculations for determining concentration are based off of Intensity Ratio (IR). The IR is defined as the background corrected intensity signal of the analyte line (Ia) divided by the internal standard value (Iis). IR = I[a]/I[is]

16. For PW and EW samples, an ICV2 will be analyzed immediately after the initial ICV. The average of the six combined burns will be used with a requirement of ±5% accuracy and an RSD of <3%.

NOTE: All samples requiring postspikes will be postspiked at 2 times the CRQL or approximately 2 times the indigenous level of the sample as required by ILMO5.2.

B. When complete, check the following:

1. The beginning and end of the raw data are signed and dated by the reviewer and verifier. These signatures indicate that the person has reviewed or verified the entire run.

2. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms. Any dilutions required have been calculated correctly and added to the reread/redigestion form. Specific instrument has been specified for client requirements if necessary.

3. Reread/redigest request forms are clipped to the front of the run.

4. For samples following Good Laboratory Practices (GLP), the raw data includes the "real-time" printout, as well as the final print file. The "real-time" printout should be signed and dated by the analyst.

5. The data are uploaded to Parallax by the reviewer and are verified from Parallax by the verifier. Correct units have been selected at upload if necessary.

C. Verification process

1. Confirm that all required pieces of QC have been uploaded to Parallax and are within specification. If there is partial QC on the current run and the samples have been analyzed more than once, check to see if there are associated runs in the hold bin waiting on additional QC to be verified.

2. Choose method of verification. (Metals verification by run, verify by multiple elements per sample, or verify by individual element.)

3. Check lab notes for each sample group.

4. Refer to the client's special requirements notebook located in the ICP office area for additional client specific information.

5. Perform any unit changes necessary.

6. If a technical decision or a client decision has been made to accept data that is not within specification, a nonconformance form must be filled out and signed by the investigator and a supervisor. Keep a copy of the form with the data for informational purposes and send the original to the Quality Assurance department.

7. After all of the elements are verified for a digest, verify the digest number and any associated tracking numbers or suite numbers.

D. Taking an instrument/analysis out of service/returning an instrument/analysis to service

NOTE: The following is taken from LOM-SOP-ES-222: In the event of an equipment failure, the following shall be performed:

1. Document the nature of the failure in the maintenance logbook

2. Document how and when the defect was discovered

3. Notification of supervisor or responsible person who can decide on appropriate action to take

4. The instrument must be clearly tagged as *Out of Service.* The tag must contain the following information:

   a. Date taken out of service

   b. Employee who took the instrument out of service

   c. Reason for tagout

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken to bring the equipment back into service.

7. Results of the corrective action (i.e., system calibration within specifications, etc.)

8. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument. This will be done on a case-by-case basis.

9. After repair, document whether the function has been fixed. Calibration or verification activities may need to be performed before the instrumentation is put back into service.

Table I

QC requirements for SW-846 6010A and 6010B ICP Metals

	Frequency	Acceptance	Corrective Action
Calibration	The calibration will contain a blank and 1 standard.		
Initial Calibration Verification (ICV)	Must be analyzed immediately following Calibration Standards.	±10% of the true value. RSD must be <5% (6010B).	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	Must be <3× IDL (6010B) Not applicable if sample concentrations are >10× the ICB (6010B). |ICB| must be <LOQ (6010A).	Data for that analyte cannot be reported from the run for the affected samples (reanalyze) (6010B). Data for that analyte cannot be reported from the run (reanalyze) (6010A).
Low Level Check (LLC)	Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB.	Use statistical limits. Not applicable if sample concentrations are >10× the true value of the LLC.	Data for that analyte cannot be reported from the run for the affected samples (reanalyze).
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA must be analyzed at the beginning and end of each run immediately following the LLC. The ICSAB must be analyzed at the beginning and end of each run immediately following the ICSA.	±20% of the true value for analytes that are spiked. |ICS| or |ICSAB| must be <2× LOQ for analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze). If an interfering element (Al, Ca, Fe, Mg) is not within specification then data for that analyte and any analyte with which it interferes cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples.	±10% of the true value. RSD must be <5% (6010B).	Data bracketing the CCV for that analyte cannot be reported (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples	Must be <3× IDL (6010B). Not applicable if sample concentrations are >10× the CCB value (6010B). |CCB| must be <LOQ (6010A).	Data bracketing the CCB for that analyte cannot be reported for the affected samples (reanalyze) (6010B). Data bracketing the CCB for the affected analyte cannot be reported (reanalyze) (6010A).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	|PB| must be <LOQ. Not applicable if analyte reading in the sample is > 20× the PB reading or <LOQ.	Redigest all associated samples.

Table I – Continued

	Frequency	Acceptance	Corrective Action
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, whichever is tighter for water LCSs. Use statistical limits for soil LCSs. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20% whichever is tighter for water LCSs. Use statistical limits for soil LCSs. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken.	Redigest all associated samples.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±25% (6010B)/±20% (6010A) whichever is tighter. RPD must be <20%.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	If the samples are >2× the LOQ the RPD must be <20. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ. Not applicable if both samples are <LOQ.	Data is flagged in the QC Summary and/or in the data package.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike(s) are not within specification.	±25% of the true value.	The data is reported in the data package.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50× the MDL.	The percent difference must be <10%.	The data is flagged in the data package.

Table II

QC requirements EPA-600/R-94/111(PW, EW) and EPA-600/4-79-020 Revised 1983 (WW) ICP Metals

	Frequency	Acceptance	Corrective Action
Calibration	The calibration will contain a blank and 1 standard.		
Initial Calibration Verification (ICV) ICV2 (for PW, EW samples)	Must be analyzed immediately following calibration. ICV2 must be analyzed immediately after the ICV to attain the average of six replicates.	±5% of the true value. RSD must be <3% (PW, EW).	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be < 3x IDL Not applicable if sample concentrations are >10x the ICB.	Data for that analyte cannot be reported from the run (reanalyze).
Low Level Check (LLC)	Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB.	Use statistical limits. Not applicable if sample concentrations are >10× the true value of the LLC.	Data for that analyte cannot be reported from the sample (reanalyze).
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA must be analyzed at the beginning and end of each run immediately following the LLC. The ICSAB must be analyzed at the beginning and end of each run immediately following the ICSA.	± 20% of the true value for analytes that are spiked. |ICSA| or |ICSAB| must be <2× LOQ for analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze). If an interfering element (Al, Ca, Fe, Mg) is not within specification then data for that analyte and any analyte with which it interferes cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples.	±10% of the true value (PW, EW). RSD must be <5% (PW, EW). ±5% of the true value (WW).	Data bracketing the CCV for the affected analyte cannot be reported (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples.	|CCB| must be < 3x IDL Not applicable if sample concentrations are >10x the CCB.	Data bracketing the CCB for the affected analyte cannot be reported (reanalyze).

Table II – Continued

	Frequency	Acceptance	Corrective Action
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	|PB| must be <LOQ. Not applicable if analyte reading in the sample is >10× the PB reading or <LOQ.	Redigest all associated samples.
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	Use statistical limits or the method limit of ±15% (PW, EW)/ 20%(WW) whichever is tighter. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 10 samples or less.	Use statistical limits or the method limit of ±15%(PW, EW)/ 20%(WW) whichever is tighter. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ.
Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	Use statistical limits or the method limit of ±30%(PW, EW)/ ±20% (WW) whichever is tighter.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	If the samples are >2× the LOQ the RPD must be <20. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ. Not applicable if both samples are <LOQ.	Data is flagged in the QC Summary and/or in the data package.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	±15% of the true value (PW, EW). ±10% of the true value (WW).	Data is reported in the data package.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50× IDL.	The percent difference must be <10% (PW, EW). The percent difference must be <5% (WW).	Data is flagged in the data package.
Samples		Results must be < Calibration range.	Sample is diluted and reanalyzed.

Table III
QC requirements CLP 4.0 and 5.2
ICP Metals

	Frequency	Acceptance	Corrective Action
Calibration	The calibration will contain a blank and 1 standard.		
Initial Calibration Verification (ICV)	Must be analyzed immediately following calibration.	±10% of the true value.	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be <CRDL (ILMO4.0). |ICB| must be <CRQL (ILMO5.2).	Data for that analyte cannot be reported from the run (reanalyze).
CRI	Must be analyzed every 20 analytical samples and at the beginning and end of each run before the ICSA and ICSAB.	±50% of the true value (ILMO4.0). ±30% of the true value except Sb, Pb and Tl (ILMO5.2). ±50% of the true value for Sb, Pb and Tl (ILMO5.2).	Data for that analyte cannot be reported from the run (reanalyze).
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA and ICSAB must be analyzed every 20 samples and at the beginning and end of each run.	±20% of the true value for analytes that are spiked. |ICSA| or |ICSAB| must be <2× the CRDL (ILMO4.0)/CRQL (ILMO5.2) for analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze). If an interfering element (Al, Ca, Fe, Mg) is not within specification then data for that analyte and any analyte with which it interferes cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples.	±10% of the true value.	Data for that analyte cannot be reported from the run (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples.	|CCB| must be <CRDL (ILMO4.0). |CCB| must be <CRQL (ILMO5.2).	Data for that analyte cannot be reported from the run (reanalyze).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	|PB| must be <CRDL (ILMO4.0). |PB| must be <CRQL (ILMO5.2). Not applicable if sample concentrations are >10× the PB value or <LOQ.	Redigest all associated samples.

Table III – Continued

	Frequency	Acceptance	Corrective Action
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Must be ±20% for water batches. Must be within statistical window for soils. If the LCS is out of specification high and the sample result is less than the CRDL/CRQL the data can be taken.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the CRDL/CRQL.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less	Must be ±20% for water batches. Must be within statistical window for soils. If the LCS is out of specification high and the sample result is less than the CRDL/CRQL the data can be taken.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples that are greater than the CRDL/CRQL.
Matrix Spike (MS)	Must be prepped of a frequency of 1 per analytical batch of 20 samples or less.	Must be ±25%.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed.
Duplicate (D)	Must be prepped of a frequency of 1 per analytical batch of 20 samples or less.	If the samples are >2× the CRDL/CRQL the RPD must be <20. If either the sample or duplicate is <5× the CRDL/CRQL the difference between the two values must be <CRDL/CRQL. Not applicable if both samples are <CRDL/CRQL.	The data is flagged in the QC Summary and/or in the data package.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	N/A	Data is reported in the data package.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50× the IDL (ILMO4.0) or MDL (ILMO5.2).	The percent difference must be <10%.	Data is flagged in the data package.

Appendix I

Definitions and explanations of the codes and symbols used on the raw data. Each heading listed below corresponds to an area labeled in Figure 1.

A. Sample table information

1. The run number.
2. The page number.
3. The tube number.
4. The sample number.
5. The first and second asterisks denote whether the sample is a background (U*), duplicate (D*), spike (R*), MSD (M*), post-digestion spike (UP), serial dilution (UL), or not a QC sample (**).
6. The weight to volume or volume to volume digestion ratio – The first number is the sample amount, the second number is the final digest volume.
7. The dilution factor – If the digest solution was diluted prior to analysis, the factor is listed here. An undiluted sample is labeled DF1.
8. Digestion batch number – Set at the time of the digestion, this number is used to track samples and QC prepared together.
9. The protocol by which the data should be reviewed (CLP, SW-846, EPA-600, etc.).

The above information (Items A.4. to A.9.) is entered into the sample table by the analyst prior to the analysis.

10. Date and time of the sample injection into the instrument.
11. The ICAP identification number.

Appendix I – Continued

B. The ICP scans all of the elements listed simultaneously during the analysis. The QC review lists the all the samples on the run. The QC review will list elements verified, good phantom, and elements that are bad (needs reread for run or batch QC). The verifier will document on the QC review if any element(s)/sample(s) were selected/deselected.

C. Two error codes in the ICP Manager software may appear here.

1. S = Saturation – The concentration of the element is greater than the photomultiplier tubes capacity to read it.

2. K = The Elements Affected by a Saturated Element – The concentration listed is not accurate, and a more accurate result can be obtained by running the sample at a dilution.

D. The concentration of each element in mg/L is listed here. The result is the average of three integrations. The %RSD of the three trials is also listed here. If the RSD is >20% for readings >2× the detection limit, the samples must be reanalyzed.

E. This section shows the individual readings in mg/L.

Figure 1

2. Page 30 of 66

LANCASTER LABORATORIES

1. Run Name: 0526701T62 — 11. Instrument ID: 05936
3. Tube: 29 — 10. Date/Time: 09/24/2005 12:08
4. Sample Number: 4608679 — 5. Class: D*** — 8. Batch: 052661848005
Initial Vol: 50.00 ← 6. → Final Vol: 50.00 — 7. DF: 1.00 — 9. Protocol Symbol: S

B. ELEMENT	C.	D. AVG (ppm)	%RSD	E. INTEGRATIONS #1	#2	#3	Re-read Re-digest
AG		0.00059	36.802	0.00036	0.00084	0.00079	
AL		0.14766	33.646	0.16032	0.09287	0.18979	
AS		0.00033	484.731	-0.00045	-0.00074	0.00218	
B		0.00608	15.041	0.00713	0.00550	0.00560	
BA		0.02192	3.559	0.02201	0.02110	0.02265	
BE		0.00222	32.133	0.00248	0.00141	0.00276	
CA		11.28918	3.663	11.37896	10.83811	11.65047	
CD		-0.00008	129.556	0.00004	-0.00014	-0.00014	
CO		-0.00074	167.730	0.00041	-0.00204	-0.00057	
CR		-0.00012	241.451	-0.00041	-0.00012	0.00017	
CU		-0.00308	15.688	-0.00359	-0.00256	-0.00308	
FE		0.08800	9.860	0.08568	0.08072	0.09760	
K		1.06992	3.089	1.07875	1.03336	1.09765	
MG		2.74134	3.349	2.75438	2.64372	2.82592	
MN		0.02083	3.828	0.02103	0.01995	0.02150	
MO		-0.00043	444.324	-0.00202	0.00168	-0.00095	
NA		9.37623	3.223	9.42690	9.05187	9.64992	
NI		0.00000	020.494	-0.00119	0.00165	-0.00047	
PB		0.00306	145.139	0.00715	-0.00167	0.00370	
SB		0.00402	56.748	0.00536	0.00533	0.00139	
SE		-0.00076	81.232	-0.00115	-0.00005	-0.00109	
SN		-0.00128	181.044	0.00108	-0.00357	-0.00136	
SR		0.05000	3.662	0.05039	0.04801	0.05161	
TI		-0.00003	146.353	-0.00001	-0.00007	0.00000	
TL		-0.00522	147.775	-0.00467	0.00221	-0.01321	
V		0.00072	52.951	0.00086	0.00029	0.00102	
Y		38731.33333	0.000	38380.00000	40084.00000	37730.00000	
ZN		0.00235	15.328	0.00249	0.00262	0.00194	

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Level 3 Document

Maintenance for the Perkin Elmer Elan 9000 ICP-MS

Reference:

Perkin Elmer Elan 9000 Hardware Guide, 2001

Cross Reference:

The following procedure is cross-referenced in this document:

Document	Document Title
SOP-IO-011	Inorganic Analysis Safety and Waste Handling Procedures
SOP-IO-034.	Operation of the Perkin Elmer Elan 9000 ICP-MS

Scope:

This procedure will describe the steps involved in the maintenance of the Perkin Elmer Elan 9000 ICP-MS.

Personnel Training and Qualifications:

1. Review and understanding of this procedure.
2. Trainee observing trained analyst performing the procedure.
3. Trainer observing trainee performing the procedure.
4. Documentation of critical steps in the training procedure.
5. Demonstration of proficiency by being able to independently maintenance the ICP-MS.

Safety Precautions:

Normal laboratory safety practices should be observed, e.g. lab coats and safety glasses. Any acid waste created during cleaning and/or sonicating should be disposed of as per SOP-IO-011.

Apparatus and Reagents:

1. Ultrasonic bath.

2. Concentrated nitric acid, Baker Instra-Analyzed or equivalent. Dilute down to %1 or %2.5 as needed.

Procedure:

Routine Maintenance:

NOTE: For more details on procedures in this document please refer to the Elan 9000 Hardware Guide. The Elan 9000 Hardware Guide also contains more detailed pictures than this document.

Documentation:

Any adjustment to an instrument, replacement of parts, etc., must be documented in the appropriate instrument logbook.

A. Remove and clean the sample introduction system when instrument performance declines.

    1. Remove spray

        a. Remove dust cover located above spray chamber

        b. Disconnect sample capillary tubing and argon gas tubing from nebulizer.

c. Unscrew spray chamber retaining ring completely (plastic knurled ring) and remove spray chamber from torch assembly.

d. Loosen two knurled screws and remove nebulizer from spray chamber.

e. To clean spray chamber, remove drain tubing and rinse with deionized water.

f. Clean nebulizer by sonicating in 1%HNO3 for a few minutes.

2. Removing and cleaning the torch

a. Use Allen wrench to push release mechanism on interface lever. Open interface by moving lever fully to the right.

b. Remove torch box cowling by loosening the two black knurled knobs. Set aside. (Caution, there is an electrical connection, cowling cannot be removed, just moved out of the way.)

c. Remove gas line connections from the torch by loosening the Swagelock fittings a few turns. Then slide the fittings and tubing up and away from the torch.

d. Rotate the torch mount (large knurled metal ring) an eighth of a turn counterclockwise, and remove entire torch assembly from spray chamber side.

e. Separate the torch from the adapter. And pull torch tip out of the adapter.

f. Sonicate torch in 1%HNO_3 for a few minutes. Rinse with deionized water.

g. Inspect tip. Sonicate along with torch if necessary. Rinse with deionized water.

h. Replace torch and/or tip if either shows excessive wear, or build-up that can not be removed.

3. Reassemble sample introduction system

a. Slide tip back into adapter. Next slide torch onto adapter. Both pieces should slide in until they hit a stop.

b. Feed torch through hole on right side of instrument. Rotate torch assembly an eighth of a turn clockwise until bayonet mount is fully seated.

c. Slide both gas fittings and tubing onto torch ends. Fittings are marked 'B' for the back fitting and 'F' for the front fitting. Tighten finger tight.

d. Reinstall torch box cowling. Both black knurled knobs should be snug, but not over tightened.

d. Place torch alignment tool on torch so that it buts up against the outermost RF coil.

e. Loosen the torch locking collar (smaller metal knurled ring) until torch moves freely. Then align the torch so that it is even with the outermost edge of the alignment tool. Retighten torch locking collar.

f. Slide tool out approx. ½ inch. Then close the interface by moving the lever full to the left.

g. Open the interface by moving the lever fully to the right. The edge of the torch should be aligned with the 5.5 mm cutout of the alignment tool. (If not, refer to Elan 9000 hardware guide)

h. Remove alignment tool.

i. Close interface by moving lever fully to the left. (Safety interlock should click into place)

j. Reconnect nebulizer to spray chamber. Tighten both knurled screws finger tight. Align nebulizer so that the sample capillary tubing inlet will be at approx. the 2 o'clock position when installed on the torch.

k. Reconnect argon gas, sample capillary, and drain tube fittings.

l. Reconnect spray chamber to torch extension with back end of chamber angled down for proper drainage.

m. Finger tighten spray chamber retaining ring. Recheck torch alignment.

n. Optimize the procedures in section 3 of the Elan software guide.

B. Removing and Cleaning the Cones.

1. Removal of cones.

a. Turn off plasma if plasma is lit. Allow two minutes or more time for cooling and vacuum to equilibrate.

b. Open top cover. Use Allen wrench to push release mechanism on interface lever. Open interface by moving lever fully to the right.

c. Align removal/insertion tool with cutout in sampler cone. Insert and rotate a quarter turn. Pull cone forward using rocking twisting motion.

d. Reverse removal/insertion tool so pins face skimmer cone. Seat tool into holes in cone. Turn tool counterclockwise to remove cone.

2. Cleaning cones.

   a. Use Kimwipe to remove any visible obstructions. If cones still dirty go to step b. Otherwise reinstall cones. (3)

   b. Invert cone into a 10 mL beaker containing 1% HNO3 so that only the tip part of the cone is immersed. Do not immerse entire cone. Make sure all air bubbles are removed prior to sonication. Sonicate cone for 5 minutes.

   c. Remove from beaker. Rinse with deionized water. Visually check tip of cone for abnormalities.

   d. Replace any cone that appears to have an oversized or non-round tip.

   e. Visually inspect O-rings for cracks or hard spots. Replace if necessary.

3. Reinstalling cones.

   a. Using removal/insertion tool, screw skimmer cone clockwise into place.

   b. Reverse tool and install sampler cone. Cone is pressure fitted into place. Cone seats flush with interface.

   c. Move interface region back into operating mode by moving interface lever fully to the left.

   d. Restart plasma (see SOP-IO-034). Sampler cone will fully seat when vacuum pump starts.

   e. Optimize the following procedures in Section 3 of the Elan software guide.

C. Removing and Cleaning the Lens

1. Removing the Lens.

   a. Shut down plasma if lit and shut down vacuum pumps.

   b. Wait at least 15 minutes for vacuum chamber to vent.

   c. Using a Phillips screwdriver, loosen screws that hold ion optics region vacuum chamber lid in place. (right hand lid, smaller of the two). Remove lid. (May require a little prying to break seal, use caution).

   d. Detach electrical connection from lens. This is the gray ball and socket connector on top.

   e. Using 2 mm Allen wrench, loosen the two lens mounting screws. (DO NOT REMOVE SCREWS)

   f. Remove lens by rotating it slightly counter clockwise and pull it out. Remove the shadow stop by rotating counter clockwise and pull it out.

   g. Unscrew lens mount nut and remove the small insulator. Slide the lens out of the mount.

2. Cleaning the lens and shadow stop.

   a. Place lens in a beaker containing no more than 2.5% HNO3.

   b. Sonicate for no more than 5 minutes. Do Not leave then lens in the acid for more than 5 minutes.

   c. Remove lens from beaker and rinse with deionized water, methanol then deionized water again.

d. Blow dry. Reinstall.

e. If unable to sufficiently remove residue, replace with new lens.

f. Shadow stop should be cleaned by wiping off surface with 2.5% HNO3 on a cotton swab. Sonicate with lens if needed. Rinse same as lens.

3. Reinstalling Lens.

a. Place lens into lens mount.

b. Place small insulator on top of lens.

c. Tighten the lens mount nut back into place. Finger tight is sufficient.

d. Place shadow stop onto mounting screw and rotate clockwise to lock. Next place lens assembly onto mounting screws and rotate clockwise to lock. Make sure electrical connector is facing up.

e. Tighten screws.

f. Reattach electrical connector.

g. Replace vacuum chamber lid, making sure cut-out is back and to the right. Tighten screws. (Do not over tighten)

h. Restart vacuum by pressing switch on the left side of the instrument, or through the Vacuum Start button in the Instrument window of the software.

i. Once system reaches adequate vacuum and the system comes to ready, retighten screws on vacuum chamber lid.

j. Optimize the following procedures in Section 3 of the Elan software guide.

D. Replace pump tubing when it shows stretching, flat spots, or RSD's start drifting up. Inspect all tubing to insure that it is secure and in good condition. Periodically perform visual check on the oil level of vacuum pumps and the water level in the coolflow.

Monthly Preventative Maintenance:

1. Vacuum instrument air filters.

2. Vacuum coolflow air filter.

Quarterly Preventative Maintenance:

Change vacuum pump oil. Interface rough pump oil will need replaced quarterly. Turbo Backing pump may not require as frequent oil changes.

Nonroutine Maintenance.

Sections 4 and 5 of the Perkin Elmer Elan 9000 Hardware Guide contain more information on the maintenance and trouble shooting of the ICP-MS. Consult a Perkin Elmer service representative for further information on troubleshooting and maintenance.

Revision Log:

Ver. #	Effective Date	Change
01	JUN 1 9 2003	New

SOPIO033_01.DOC
060203

Prepared by: _____ 7/9 Date: 6/5/03
Senior Chemist

Approved by: Robert L. Strodel II 8" Date: 6.5.03
Metals Management

Approved by: Dorothy McClure Date: 6/5/03
Quality Assurance

Lancaster Laboratories
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Uncontrolled Copy

SOP-IO-033. 01 PA #1
Procedure Effective Date: 06/19/03
PA Effective Date: APR 2 4 2006
Page 1 of 1

Procedural Amendment #1

Procedure Title: Maintenance for the Perkin Elmer Elan 9000 ICP-MS

Reasons for addition(s) or change(s): Changing the frequency of Preventative maintenance to as needed instead of monthly or quarterly.

Samples or project affected: All

List change(s) or addition(s) (specify which section): *Monthly Preventative Maintenance and Quarterly Preventative Maintenance sections have been combined into one section, to be performed as needed, labeled "Preventative Maintenance."*

Preventative Maintenance: performed as needed

1. Vacuum instrument air filters
2. *Vacuum coolflow air filters*
3. Change vacuum pump oil
4. Replace interface rough pump oil
5. Replace turbo backing pump oil

SOPIO033_01PA1.DOC
042406

Prepared by: _____ 859 Date: 4/24/06
Specialist

Approved by: _____ Date: 4.24.06
Metals Management

Approved by: Elaine Stoltzfus Date: 4/24/06
Quality Assurance

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Operation of and Analysis with the Perkin-Elmer Elan 9000 ICP-MS

Reference:

1. Perkin-Elmer Elan Version 2.4 Software Guide, 2001.
2. Perkin-Elmer Elan 9000 Hardware Guide, 2001.
3. EPA Method 200.8, USEPA 600/R-94-111.
4. EPA Method 6020, USEPA SW-846, 12/96.
5. CLP ILM05.2, Exhibit D/ICP-MS, USEPA CLP Statement of Work.

Cross Reference:

Document	Document Title
LOM-SOP-ES-207	Establishing Control Limits
LOM-SOP-ES-212	Internal Chain-of-Custody Documentation
MC-IO-003	Fixed-Volume Hand-Held Pipettes
SOP-IO-007	Preparation of Standards and Solutions
SOP-IO-011	Inorganic Analysis Safety and Waste Handling Procedures
SOP-IO-012	Calculations Used by the Inorganics Group
SOP-IO-033	Maintenance for the Perkin Elmer Elan 9000 ICP-MS
SOP-IO-035	Quality Control Procedures for ICP-MS
Form 3865	Elemental Analysis by Elan 9000 Inductively Coupled Plasma Mass Spectrometry-SW-846
Form 3982	Elemental Analysis by Elan 9000 Inductively Coupled Plasma Mass Spectrometry-CLP

Purpose:

The purpose of this SOP is to outline the proper procedure to operate the Elan 9000 ICP-MS and the procedure for setting up and pouring an ICP-MS run.

Scope:

This procedure will cover the hardware, software, and quality assurance necessary in the operation of the Perkin-Elmer Elan 9000 ICP-MS, as well as the apparatus and quality assurance procedures needed to set up and pour an ICP-MS run.
The procedure is the same regardless of method and/or protocol.

Basic Principles:

ICP-MS is an analytical instrument that uses the energy of the inductively coupled plasma to generate the ions to be analyzed in the mass spectrometer. Several different means can be utilized for sample introduction but this system uses a computer controlled peristaltic pump that delivers the sample from the autosampler into a cross flow nebulizer attached to a Scott spray chamber.

Personnel Training and Qualifications:

1. Review and understanding of this procedure.
2. Trainee observing trained analyst performing the procedure.
3. Trainer observing trainee performing the procedure.
4. Review of trainee's data by trainer.
5. Documentation of critical steps in the training procedure.
6. Demonstration of proficiency by being able to independently run the ICP-MS.

Interferences:

ICP-MS interferences include isobaric elemental interferences and polyatomic ion interferences derived from the plasma gas, reagents, or sample matrix. Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for using internal standardization. Memory interference is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time of the autosampler tubing overcomes any memory interference.

Apparatus and Equipment:

A. The following is a list of the hardware used in the Perkin-Elmer Elan 9000 ICP-MS system. Included is a brief description of each component.

1. Spectrometer – The Perkin-Elmer Elan 9000 is an inductively coupled plasma mass spectrometer. The sample introduction system consists of a Ryton crossflow nebulizer on a Scott spraychamber attached to a concentric quartz tube plasma torch.

2. Autosampler – The Perkin-Elmer AS-93*plus* autosampler has capacity for 149 samples and 8 standards. The autosampler parameters for each automated run are entered into the Sample window of the Elan software as described in Procedure C.6.

3. Coolflow – The Polyscience cooling system is set up to deliver cooling liquid to the ICP-MS at a regulated pressure of 50 psig.

4. Computer – The Perkin-Elmer Elan 9000 is controlled by a Windows-based IBM compatible PC with Elan Version 2.4 ICP-MS software installed.

5. Vacuum Pumps – There is a rotary vane vacuum pump hooked up to the region between the sampler and skimmer cones. This pump maintains the interface region at approximately 4 torr of vacuum. In the quadrapole region, there is a dual inlet turbo molecular pump. This pump maintains the ion optic region at a vacuum of approximately 8×10^{-4} torr, and the mass filter region at a vacuum of approximately 1×10^{-5} torr.

B. The following is a list of the apparatus necessary to the setup and pouring of an ICP-MS run:

1. ICP-MS preprinted run sheets (pages 1-2)

   Page 1: Includes standards and initial QC preprinted according to method and instrument. The first tube in the sequence is 38 due to the configuration of the autosampler trays.

   Page 2: Includes tubes 69-99 and may be used for any method.

2. Test tube racks

3. 17 × 100-mm Polystyrene Tubes

4. Filter paper – Whatman No. 540, 90 mm ashless

5. 1 × 100 10-mL sterile disposable syringes

6. 25-mm syringe filters, PTFE, 0.45 µm

7. 30-mL polypropylene medicine cups

8. Eppendorf fixed-volume hand-held pipettes (50, 100, 200, 250, 500, and 1000 µL)

   NOTE: For routine operation, calibration, and maintenance of Eppendorf fixed-volume hand-held pipettes, see MC-IO-003.

9. Eppendorf pipette tips (or equivalent)

   a. 1 to 200 µL yellow or clear plastic

   b. 200 to 1000 µL blue plastic

Reagents and Standards:

Refer to SOP-IO-007, Section D.

Safety Precautions:

Refer to SOP-IO-011 for laboratory safety procedures.

Procedure:

A. Operation of the Perkin-Elmer Elan 9000

1. To operate Elan software:

   If not already active, double click on the Elan icon on the computer desktop to start the software.

2. Warm start-up of the Perkin-Elmer Elan 9000 ICP-MS:

   a. Open the Instrument window and select the Front Panel tab. Verify that the System Status is "Ready". If the System Status is "Not Ready", click on the Diagnostics tab to find which parts of the system are not operating within specs. (See Section 14 of the Elan Version 2.4 Software Guide for further use of the diagnostics.)

   b. Check that the waste drain vessel is not full, and has adequate space remaining to complete the automated run without overfilling.

   c. Check that the sample, internal standard, and drain lines are attached to peristaltic pump and that all tubing is connected properly.

   d. Check to see that there is adequate internal standard solution to complete the entire automated run.

   e. Check to see that the autosampler rinse vessel has sufficient rinse for duration of the automated run. If not, refill with 6% HNO_3 solution. Both the sample and internal standard sipper probes should be placed in the 2-L bottle filled with deionized water.

   f. Open the Instrument window and select the Front Panel tab. Check the vacuum pressure. It should read between 1×10^{-6} Torr and 2×10^{-6} Torr. Pressure significantly lower or higher may indicate an instrument hardware problem. Once you have verified that the pressure is at an acceptable level, record the pressure reading as the Base Vacuum Pressure. Click on the "PLASMA START" button on the Front Panel tab. The plasma should ignite within 20 seconds. After the plasma has ignited, open the Devices window and set the peristaltic pump speed to -24 rpm. Recheck the vacuum pressure and record the current reading as the Running Vacuum Pressure. If plasma fails to ignite, refer to Elan Software Guide section 2 "What to Do if Ignition Is Unsuccessful."

g. Allow plasma to warm up for 10 to 15 minutes if system has been down for 15 minutes or less. Allow at least 30 minutes of warm up time if system had been down for more than 15 minutes. To verify that the instrument has fully warmed up select the Diagnostics tab in the Instrument window. The "Main water temp" should be approximately 38°C; the "Interface water temp" should be approximately 50°C; and the "Torch box temp" should be approximately 47.5°C.

These temperatures are given as guidelines. The actual temperatures may vary by 2 or 3 degrees depending on ambient conditions and other factors. These temperatures, however, should always reach a steady state before beginning daily tuning and analysis.

3. Cold start-up of the Perkin-Elmer Elan 9000 ICP-MS

a. This procedure is used when the system has been down for an extended time period and the vacuum pumps have not been running.

b. Check oil levels in both vacuum pumps before proceeding.

c. Refer to Section 2-3 of the Elan Software manual for detailed instructions for starting up the instrument.

B. Setting up an ICP-MS run

1. Obtain appropriate preprinted ICP-MS run sheets (based on method) and record the initials, employee number, and date the run was set up.

2. Determine the batches to be analyzed.

NOTE: An ICP-MS run typically contains no more than 60 tubes.

3. The run sheets should then be filled out with the sample name, class, initial volume (IV), final volume (FV), dilution factor (DF), batch number, protocol, SDG, and comments. This information can be found on the Prep Batch Sheet.

a. Sample names include:

(1) PBW – Prep blank (water)

(2) LCSW – Laboratory control sample (water)

(3) LCSDW – Laboratory control sample duplicate (water)

(4) PBS – Prep blank (solid)

(5) LCSS – Laboratory control sample (solid)

(6) LCSDS – Laboratory control sample duplicate (solid)

(7) Lancaster Laboratories' sample number

(8) CCV – Continuing calibration verification

(9) CCB – Continuing calibration blank

(10) CRI – Contract-required detection limit standard

(11) LLC – Low level check

(12) ICSA – Interelement correction standard – A

(13) ICSAB – Interelement correction standard - AB

(14) S0 – Calibration blank

(15) S1, S2, S3 – Calibration standard 1, 2, 3

NOTE: For definition of above, see SOP-IO-014.

b. Initial volume (IV) – The sample aliquot digested.

c. Final volume (FV) – The final sample volume after digestion.

d. Dilution factor (DF) -- The dilution factor of the sample prepared at the time of analysis. Needed to bring the sample into the linear range of the instrument, to negate a matrix effect, or for serial dilutions.

e. Batch No. – The batch number of the sample. By convention, the batch number is recorded opposite the first tube listed for the batch as well as the first line of each additional page.

f. Protocol – The protocol used to review the data for specific method requirements in the IDAT database.

g. SDG – Sample delivery group number for data package samples.

h. Comments – Any description of the sample (from prep logs), status of the sample (i.e., RUSH, Promised), and chain-of-custody documentation needed, if any, should be recorded here. Reread elements are also recorded here. Lot numbers for standards must also be recorded here.

4. When setting up a run, Batch QC (i.e., PB, LCS, background, duplicate, matrix spike, matrix spike duplicate, post-digest spike, and serial dilution) should be placed in the same block of ten or fewer samples. If there are two LCSs, they should be placed one after the other.

   a. ICV/ICB must be analyzed immediately after the calibration curve.

   b. CCV/CCB must be run after every ten analytical samples.

   c. LLC or CRI, ICSA, ICSAB, CCV, CCB immediately follow the ICV/ICB and must conclude each run. For CLP 5.2, there must be a CRI, ICSA, ICSAB, CCV, and CCB following every 20 analytical samples.

   d. Any deviations from protocol should be noted in the Comments Section of the cover page.

   e. Any unused portion of the run sheet must be "Z'd" out.

C. *Setting up Elan software for automated analysis*

1. Open the Elan software if not already open.

2. Daily Tuning.

   a. After the instrument has been allowed to warm up, choose "Open Workspace" from the "File" menu and open "Daily Tuning.wrk."

   b. Aspirate the Elan 6100 Setup/Stab/Masscal Solution (10 ppb Mg, Cu, Rh, Cd, In, Ba, Ce, Pb, and U). Be sure that both the sample probe and internal standard probe are placed into the solution. Allow the solution to flush until all air bubbles have traveled through both sample and internal standard lines. Cu, Ba, and Cd are not used in the evaluation of the instrument tuning.

   c. After 10 seconds of additional flush time, make sure the "Peak Width Only" box is checked and click "TUNE MASS SPEC." Read 5 replicates of the tuning solution. Widths should read back between 0.64 and 0.66 amu. Verify that the Measured Mass is within ±0.1 amu of the Exact Mass. If not, perform a Mass Calibration (refer to the Elan version 2.4 Software Guide, p. 3-16). A Mass Calibration should only be performed after any instrument maintenance issues have been addressed (refer to SOP-IO-033). Check the Summary section and verify that the RSD is less than 5 for masses 3, 24, 103, 115, 140, 208, and 238. The Daily Tuning check shall be considered acceptable only when the Peak Width, Measured Mass, and RSD meet the specified criteria.

   d. If peak widths fall outside of this range, they may be adjusted by changing the RDAC Value. To lower the measured peak width by 0.01, raise the RDAC Value by 3 and vice versa. For He only: raising the RDAC Value by 3 will lower the peak width by ~0.02. The new RDAC values may be quickly determined by using the Excel spreadsheet "Tuning Calculator" located in K:\SHRDATA\ICP-MS.

Enter the Measured Peak Width values from the Tuning window into the "Measured Peak Width" column in the Excel spreadsheet. Enter the values from the "New DAC Value" column (in the Excel spreadsheet) into the appropriate RDAC Value cell in the Tuning window. After changing the appropriate RDAC values, save the tuning file (select "File"→ "Save") and click "TUNE MASS SPEC." Repeat this process until all widths read back between 0.64 and 0.66 amu. Always save the tuning file before proceeding to Daily Performance. **NOTE:** this check is performed once at the beginning of each day OR if maintenance is performed this check is done after maintenance, before starting any analytical runs (even if Tuning has been done earlier on the same day).

3. Once daily tuning passes criteria, chose "Open Workspace" and open "Daily Performance.wrk." Continue to aspirate the Elan 6100 Setup/Stab/Masscal Solution. Type "Daily Performance" in the Sample Name field and click "ANALYZE SAMPLE." Verify that the Daily Performance results are within specification (see Attachment I). If criteria are not met for any parameter refer to Elan Software Guide section 3 "Tuning and Optimization" for corrective actions. **NOTE:** this check is performed once at the beginning of each day OR if maintenance is performed this check is done after maintenance, before starting any analytical runs (even if Daily Performance has been checked earlier on the same day).

4. After Tuning and Daily Performance checks are complete, place the Instrument Tuning Report and the Daily Performance Report in the "Tuning/Daily Performance Reports" folder. In the Method window select the Sampling tab. Click the "PROBE" button. The "Autosampler Probe Control" window appears. Click the "GO TO RINSE" button, then click "OK." This will return the autosampler arm to the rinse station and start the pump for the instrument rinse. Place the internal standard probe in the internal standard solution and place the sample probe into the rinse station (through the autosampler arm). If either Tuning or Daily Performance fail see the Elan Version 2.4 Software Guide and/or the Elan 9000 Hardware Guide for troubleshooting the problem.

5. Before you begin each analytical run, you will need to print a copy of the most recent Instrument Tuning Report and Daily Performance Report for that day. These will be kept with the analytical run. To reprint the Instrument Tuning Report, open the Daily Tuning workspace ("Daily Tuning.wrk"), select the Dataset window, highlight the last row of data, and click the "REPROCESS" button. To reprint the Daily Performance Report open the Daily Performance workspace ("Daily Performance.wrk"), select the Dataset window, highlight the last row of data, and click the "REPROCESS" button. Verify that both match the most recent reports (for that day) that were placed in the "Tuning/Daily Performance Reports" folder.

6. Chose "Open Workspace" from the "File" menu and open "default.wrk" (This is the default workspace for sample analysis; other workspaces may be used if appropriate). Select the Sample window. Enter the appropriate sample info into the Sample window. The "Sample Template" button can be used for entering incremental sample numbers. Under Description enter the batch number (i.e., 032196050003), sample class (i.e., U***), protocol, and dilution factor (i.e., 5), and protocol separated by commas. For example: 032196050003,U***,3,5. Verify that the flush/read/delay times and speeds are set correctly. "Sample Flush(sec)" should always be set to 30; "Read Delay(sec)" should always be set to 20; "Sample Flush Speed" and "Wash Speed" should always be set to -48; "Delay & Analysis Speed" should always be set to -24; "Wash(sec)" (i.e. rinse time) can be adjusted depending on the sample matrix, but should be the same throughout the entire run. A rinse time of 60 seconds is usually sufficient for most samples. The "Edit"→ "Fill Down" command (Ctrl-F) can be used to speed up the entry of sample info. To use this function, highlight the row you wish to copy along with the rows beneath it that you wish to copy to. Press Ctrl-F to copy the values from the top row to the ones below it. If the run will end during your shift set "Wash(sec)" value to –24 for the last tube in the run sequence. Otherwise set it to –1 (this is to reduce the consumption of Internal Standard solution when no one is present to remove the sipper from the internal standard solution at the end of a run). When all info is typed into the Sample window save the file using "File"→ "Save" (the default file name is "default.sam").

7. Select the Dataset window. Under "File" click "NEW". Use the IAG run #, minus the instrument name, as the new Dataset name (i.e., 0323504, where 03 is the two-digit year, 235 is the Julian day number, and 04 is the run number for that day). Type the appropriate run # in the "File Name" field and click "OPEN."

8. Select the Method window. Click the "REPORT" tab on the right hand side of the Method window. Under the section titled "Report to File", change the Report Filename to the appropriate IAG run # (i.e., c:\import\0320603.E01). The file name should end in ".E01." Save the Method window Under "File"→ "Save" before proceeding.

9. The Blank (S0) goes in pos. 38, S1 in pos. 39, S2 in pos. 40, and S3 in pos. 41. Check standards and samples should start at pos. 42.

10. Place the sample tray containing the samples and standards to be analyzed in the correct autosampler tray position. Open the Calibration View window and open a new calibration file using "File"→ "New." If prompted to save the calibration file select "No." If prompted to clear blank and remove calibration information, always select "Yes." Verify that all sample info is typed in correctly in the Sample window. Highlight all samples to be run, and click "ANALYZE BATCH." The system will run the Blank, calibration standards, and samples. If prompted to clear QC data and/or all existing Blank/Calibration data, always select "Yes."

11. After a run is complete: to conserve instrument rinse and reduce acid waste production open the Method window, select the Sampling tab, and click the "PROBE" button. When the Autosampler Probe Control window appears, click the "GO TO STANDBY" button and then click "OK." This will move the autosampler arm to the standby position and turn off the instrument rinse pump.

D. Pouring an ICP-MS run

It is important to minimize any chance of contamination, both of yourself and the samples. Keep your hands and the work area clean at all times. Wear appropriate PPE at all times (see SOP-IO-011).

1. Choose the appropriate run sheet. Record on each page of the ICP-MS run sheet: initials, employee number, and the date.

2. Prepare and record any calibration standards, check standards, and other solutions needed to complete the pouring of the run.

   NOTE: See SOP-IO-007, Section D.

3. Obtain the appropriate number of tubes, number each tube (the first tube number is 38), and place them in test tube racks. If you are using one of the autosampler racks it is not necessary to number the tubes; instead pay close attention to the numbering system on the rack, as it corresponds with the numbering system on the preprinted run sheets.

4. Pour appropriate standards and initial run QC as labeled on the run sheet (approximately 5 mL for ICP-MS). Document lot numbers of the standards in the comment section.

   Any samples that require chain of custody may be taken out of the locked storage cabinet. The transfer should be documented on the chain-of-custody form with first initial, full last name, employee number, date and time (military). Samples must be in the analyst's possession until they are signed back into custody of locked storage. See LOM-SOP-ES-212.

5. Prepare and label the PDS required for each batch (sample volume permitting). A PDS is prepared by placing 0.1 mL of a custom ordered PDS solution into a 5 mL volumetric and bringing to volume with 4.9 mL of background sample. Record the lot number of the PDS solution in the comments column of the ICP-MS run sheet.

6. Prepare a serial dilution by diluting the background sample approximately 5×. If the background sample chosen for serial dilution has been diluted due to matrix interference or to bring the analyte concentration into the linear range of the instrument, the diluted sample must be diluted 5× (i.e., if Bkg = DF5, S.D. must = DF25). Document how the dilution(s) was/were made in the comment section.

7. Samples that need to be filtered that were digested using the hotblock tubes filter those samples that are cloudy or contain particulate with the hotblock filters. Samples that are digested in beakers or the soil digestion vessels may be filtered using a 10-mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter. If any samples are filtered, the prep blank and LCS must also be filtered. Document all filtrations on the run cover sheet.

8. Pour each sample or sample filtrate into the appropriate tube. Usually, the order of the batch QC is PB, LCS, (LCSD), Bkg, PDS, DUP, MS, (MSD), and SD.

9. Cover the run with plastic wrap to prevent contamination of the samples.

10. Return samples to sample storage. If samples require chain of custody, return samples to locked storage and sign the COC.

11. **NOTES:**

    a. A post-digest spike and a serial dilution will be performed on one sample in each digestion batch. Typically, the background sample is chosen. If the batch QC is split between two samples, the post-digest spike will be performed on the background sample accompanied by a matrix spike; the serial dilution will be performed on the background sample accompanied by a matrix duplicate. (For definition of batch QC, see SOP-IO-014, pages 1 and 2). If sample volume is limited, the duplicate may be used for the PDS and SD.

    b. Batches with only field blanks or equipment blanks do not need a post-digest spike or a serial dilution.

c. Air filter batches need only a serial dilution on one sample in the batch (a post-digest spike is not required).

d. "As Received" samples should be run with a blank and LCS, LCSD (prepared by the analyst). These "batches" are recorded in the Dilute & Run Batch Book located in the ICP lab.

e. After the run has started, note the run number on the batch sheet, copy the batch sheet at 65% reduced size, file the original copy in the batchbook, and write the batch number on the top of the reduced copy in Sharpie to keep with the samples. Write the run number on any reread sheets and file the reread sheet in the reread book.

f Documentation is of utmost importance. Double check all entries.

Calculations:

Refer to SOP-IO-012.

Statistical Information:

Refer to LOM-SOP-ES-207.

Quality Assurance/Quality Control:

Refer to SOP-IO-035 for acceptance criteria for reviewing an ICP-MS run.

Revision Log:

Ver. #	Effective Date	Change
01	06/26/03	New
02	12/26/03	• Changes in wording and clarification of procedures throughout the document. • Updated to Level 3 format

Ver. #	Effective Date	Change
03	03/24/04	Major changes are as follows: • Procedure A.2.g. – Incorporated Procedure Amendment • Procedure C.2.c. – Incorporated Procedure Amendment • Procedure C.5. – Incorporated Procedure Amendment • Procedure B.3.a. (14, (15), (16) – Removed HS1, HS2 – High standards and renumbered remaining entries. • Cross Reference – Added Form 3982
04	11/17/04	Major changes are as follows: • Updated section C.2.c
05	APR 1 4 2006	Major changes are as follows: • Incorporated procedural amendment: C.2.b – Added "Cu, Ba, and Cd are not used in the evaluation of the instrument tuning • B.3.f – Changed the IAG reference to IDAT • D.7 – Changed the filtering information for samples that need filtered.

SOPIO034_05.DOC
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Prepared by: _____ Date: 3/23/06
Specialist

Approved by: _____ Date: 3.27.06
Metals Management

Approved by: _____ Date: 3/31/06
Quality Assurance

Attachment I

Daily Performance Acceptance Criteria

In Sensitivity	>300,000 cps
Mg Sensitivity	>40,000 cps
Pb Sensitivity	>100,000 cps
CeO/Ce	≤0.03
Ba++/Ba+	≤0.03
Background	<30 cps @ Mass 220
Net RSd	≤%5.0

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Uncontrolled Copy

SOP-IO-035.09
Supersedes Date: 07/11/06
Effective Date: SEP 28 2006

Quality Control Procedures for ICP-MS

Approvals:

Prepared by: David E Bell — Date: 9-5-06
Chemist

Approved by: Elizabeth Strathmann — Date: 9.11.06
Metals Management

Approved by: Dorothy M Love — Date: 9/14/06
Quality Assurance

Revision Log:

Ver. #	Effective Date	Change
01	06/26/03	New
02	03/25/04	Major changes are as follows: • Definitions – Updated Method Detection Limit definition • Updated QC charts at end of SOP
03	11/18/04	Major changes are as follows: • Updated Definitions and Procedure Sections
04	01/31/05	Major changes are as follows: • Table I – Updated calibration and sample requirements
05	10/20/05	Major changes are as follows: Added method and mass chart to the procedure section of SOP.
06	11/18/05	Major changes are as follows: • Updated Procedure section – A.; B.3. • Updated Tables II and III
07	02/14/06	Major changes are as follows: • Updated Cross Reference, Personnel Training and Qualifications, and Procedure sections
08	07/11/06	Major changes are as follows: • Clarified item 23. in Definitions section regarding LDR and the use of the LRS standard • Updated table in Procedure section A. • Added procedures for evaluating mass interference and evaluating the use of uncorrected data in Procedure section B. • Updated Tables I, II and III
09	SEP 28 2006	Major changes are as follows: • Updated Tables I, II and III • Moved Approval and Revision Log sections to beginning

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Reference:

1. ILM05.2, Exhibit D/ICP-MS, USEPA CLP Statement of Work.
2. Method 6020, USEPA SW-846, 12/96.
3. Method 200.8, USEPA 600/R-94-111.

Cross Reference:

Document	Document Title
LOM-SOP-ES-207	Establishing Control Limits
LOM-SOP-ES-222	Instrument and Equipment Maintenance and Calibration
SOP-IO-007	Preparation of Standards and Solutions
SOP-IO-012	*Calculations Used by the Inorganics Group*

Purpose:

This SOP is designed to provide consistent guidelines for the evaluation of ICP-MS data.

Scope:

This procedure applies to analyses performed in Environmental Sciences using ICP-MS for identification and quantitation of metallic constituents.

Definitions:

Batch and instrument QC

1. Analytical Batch – A group of field samples that are digested and analyzed together. A batch consists of no more than 10 samples for EPA 600 methods or no more than 20 samples for other methods.

2. Analytical Samples – Analytical sample is defined as any solution introduced into an instrument on which an analysis is performed, excluding instrument calibration, ICV, ICB, CCV, CCB, and tunes. Analytical samples include undiluted and diluted samples, matrix spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, ICSs, CRIs, LLCs, PBs, LCSs, PEs, and Linear Range Samples (LRSs).

3. Background Sample (U) – The original sample from which the batch QC is derived. The background sample is either site specific or randomly selected.

4. Continuing Calibration Blank (CCB) – A reagent blank run immediately after every CCV. This is used to monitor the stability of the low end of the calibration.

5. Continuing Calibration Verification (CCV) – A mid-range standard run at a frequency of 10% (every ten samples) throughout the run. This is used to monitor instrument drift.

6. Contract Required Detection Limit (CRI) – A standard analyzed at the Contract Laboratory Program (CLP) required detection limit. This standard must be at the beginning of each sample analysis run, but not before the ICV/ICB. This sample verifies linearity near the limit of quantitation. ILM05.2 requires the CRI to be analyzed every twenty analytical samples.

7. Duplicate Sample (D) – A replicate of the original sample, processed in parallel. This sample is used to provide a measure of the in-lab repeatability (precision) of the analytical process. The duplicate sample is either site specific or randomly selected.

8. Initial Calibration Verification (ICV) – This is a standard near the middle of the calibration range, prepared from a different source than the calibration standards. It is used to prove that the instrument is calibrated correctly at the start of the run.

9. Initial Calibration Blank (ICB) – This is a standard reagent blank, used to prove that the low end of the calibration is acceptable. It must be run immediately after the ICV.

10. Interelement Correction Standard-A (ICSA) – A standard containing high concentrations of commonly interfering elements. It is used to assess the interferences due to matrix elements that can normally be expected to be found in a sample.

11. Interelement Correction Standard-AB (ICSAB) – A standard containing both interfering elements and target analytes, run immediately after the ICSA. It is used to demonstrate the effectiveness of the correction factors in use.

12. Instrument detection limit (IDL) – A value determined from analyzing 7 standard solutions (undigested) at a concentration 3× to 5× the anticipated IDL on three nonconsecutive days. The standard deviation obtained for these multiplied by 3 is the IDL. These must be performed quarterly on each instrument used for an analyte.

    Non-CLP Analyses – A value determined for the purpose of evaluating the ICB/CCBs for data package samples. It is determined by analyzing 7 standard solutions at a concentration 3× to 5× the anticipated IDL. This value is obtained annually for each element analyzed on an instrument.

13. Laboratory Control Sample (LCS) – This is a matrix-matched synthetic sample of known composition. It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes.

14. Laboratory Control Sample Duplicate (LCSD) – This is a duplicate of the matrix-matched synthetic sample of known composition. It is used to judge efficiency of the digestion procedure, as measured by the % recovery of the analytes. It is also used as a measure of the precision of the analytical process.

15. Limit of Quantitation (LOQ) – The level above which quantitative results may be obtained with a specified degree of confidence. It is based on a value 3× to 5× the MDL. CLP 5.2 samples are reported using the MDL and CRQL.

16. Low Level Check Standard (LLC) – A low-level standard used to monitor the performance of the instrument near the detection limit.

17. Matrix Spike Duplicate (MSD) – A duplicate of the Matrix Spike Sample (R) which is a replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure. It is also used as a measure of the precision of the analytical process. The matrix spike duplicate sample is either site specific or randomly selected.

18. Matrix Spike Sample (R) – A replicate of the original sample spiked with a known amount of analyte. This sample is used to determine if there are any matrix effects that could influence analyte recovery during the digestion procedure. The matrix spike sample is either site specific or randomly selected.

19. Method Detection Limit (MDL) – The minimum concentration of a substance that can be reported with 99% confidence that the analyte concentration is greater than 0. It is determined by analyzing 7 digested standards at an estimated concentration 2.5x to 5x the signal/noise ratio. MDLs are performed on all instruments used to determine each analyte. The MDLs are available in the LIMS system since they can change frequently.

20. Post Digestion Spike (PDS) – This sample is a spike of the Background Sample prepared after digestion, at the time of analysis. It is used to determine if low spike recoveries are due to problems in the digestion or are matrix related.

21. Preparation Blank (PB) – This is a reagent blank carried through the entire digestion procedure. It is used to determine if contamination has occurred during the digestion procedure.

22. Serial Dilution (SD) – This sample is a 1:4 (5x) dilution of the Background Sample, prepared after the digestion. It is used to indicate the presence of any matrix effects that could cause a nonlinear response at the instrument.

23. Linear Dynamic Range (LDR) – By default the LDR is equal to the concentration of the upper calibration standard . If a Linear Range Standard (LRS) is analyzed with the run, and recovers within the specified limits, the LDR is extended to the concentration of the LRS. If a multiple point calibration is used, the correlation coefficient for the curve must be ≥0.995.

Personnel Training and Qualifications:

1. Review and understanding of this procedure
2. Trainee observing trained analyst performing the procedure
3. Trainer observing trainee performing the procedure
4. Review of the trainee's data by trainer
5. Documentation of critical steps in the training process
6. Demonstration of proficiency by being able to independently review ICP/MS data

Procedure:

A. Masses used for ICP-MS analysis – The primary mass (designated with an asterisk) is the mass that would normally be reported. Another mass would be selected if there were interferences for the primary mass.

Isotope	Mass	Soil Analysis #	Water Analysis	Method
Be 9	9	6127	6027	A, B, C
Cr 52*	52	6131	6031	A, B, C
Cr 53	53	6131	6031	A, B, C
Ni 60*	60	6139	6039	A, B, C
Ni 61**	61	6139	6039	A, B, C
Ni 62	62	6139	6039	A, B, C
Cu 63*	63	6133	6033	A, B, C
Cu 65	65	6133	6033	A, B, C
Zn 66*	66	6149	6049	A, B, C
Zn 67**	67	6149	6049	A, B, C
Zn 68	68	6149	6049	A, B, C
As 75	75	6125	6025	A, B, C
Se 77	77	6141	6041	A, C
Se 78**	78	6141	6041	A, C
Se 82*	82	6141	6041	A, C
Ag 107*	107	6142	6042	A, B, C
Ag 109	109	6142	6042	A, B, C
Cd 111*	111	6128	6028	A, B, C
Cd 114	114	6128	6028	A, B, C
Sb 121*	121	6124	6024	A, B, C
Sb 123	123	6124	6024	A, B, C
Ba 134**	134	6126	6026	A, B, C
Ba 135	135	6126	6026	A, B, C
Ba 136**	136	6126	6026	A, B, C
Ba 137*	137	6126	6026	A, B, C
Tl 203*	203	6145	6045	A, B, C
Tl 205	205	6145	6045	A, B, C
Pb 206	206	6135	6035	A, B, C
Pb 207	207	6135	6035	A, B, C
Pb 208***	208	6135	6035	A, B, C

*Primary mass

**These isotopes are not used

***For Pb masses 206, 207 and 208 are summed for calibration and analysis

A = EPA 200.8 elements

B = EPA 6020 elements

C = CLP

Note: All elements are available for analysis by any method. Precision and accuracy data is available upon request.

B. Raw data quality checks

1. Make sure that the run is correctly labeled, dated, and signed and that the corresponding cover sheet is attached to the front of the run.

2. Verify that the appropriate Tuning Report is with the run.

3. For calculations used by the inorganics groups see SOP-IO-012.

4. For run and batch QC/Calibration frequency, acceptance criteria and corrective action, see Method Specific Tables I (EPA 200.8), II (EPA-6020), and III (CLP5.2). For information on statistical windows refer to LOM-SOP-ES-207.

5. For spike levels of run QC see SOP-IO-007, Section D.

6. For spike levels of batch QC see SOP-IO-007, Sections G and H.

7. LOQs are available to analysts in the LIMS and on charts that are updated as needed.

8. Check to make sure that all results are below the linear range limit (see the Definitions section #23). If a sample reading is above the linear range, then reread the sample at a dilution sufficient to bring the sample concentration to approximately the middle of the calibration range. For EPA 600, reread the sample at a dilution if it reads >90% of the linear range. For CLP 5.2, the diluted sample reading must fall within the upper half of the calibration range.

9. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings < (-LOQ).

10. Check for carryover between samples. Sample RSD >20%, with a concentration > the LOQ decreasing progressively over time (i.e., Reading 3<2<1). Flag any suspect samples for reread.

11. For all EW (samples from public drinking water sources), check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once so that the supplier can be notified. Suppliers must be notified within 1 hour.

Analyte	MCL (mg/L)
Sb	0.006
As	0.05
Ba	2 (1)**
Be	0.004
Cd	0.005
Cr	0.1 (0.05)**
Se	0.05 (0.01)**
Tl	0.002
Al*	0.2
Cu*	1.0
Fe*	0.3
Mn*	0.05
Ag*	0.1 (0.05)**
Zn*	5.0

***Secondary regulated contaminants**

****The federal MCLs for these analytes are greater than Pennsylvania MCLs. The numbers in parentheses are the MCLs effective in Pennsylvania**

12. For analyte readings greater than 2 x LOQ verify that the RPD between the primary and secondary isotopes is less than 20% (for analytes with multiple isotopes).

13 For Arsenic readings greater than 2 x LOQ verify that there is not significant interference at mass 82 (i.e. Se 82 > 2 x LOQ, Se 82 > Se 77, and the RPD between Se 82 and Se 77 is > 20%). If so, evaluate whether reporting from the uncorrected Arsenic line (xAs 2) is appropriate. Uncorrected As should only be reported when all of the following conditions are met:

a. The corrected As line, "As", is greater than 2 x LOQ.

b. The corrected As line, "As", is greater than the uncorrected As line, "xAs 2".

c. The RPD between the corrected As line, "As", and the uncorrected As line, "xAs 2", is greater than 20%.

14. For Cadmium readings that are less than the negative LOQ evaluate whether reporting from the uncorrected Cadmium line (xCd 111) is appropriate. In this case, uncorrected Cd should only be reported when all of the following conditions are met:

    a. The corrected Cd line, "Cd", is less than the negative LOQ.

    b. The amount of Molybdenum in the sample is insignificant (i.e. less than the ICSA).

    c. The uncorrected Cd line, "xCd 111" does not itself read less than the negative LOQ.

15. For Cadmium readings greater than 2 x LOQ evaluate whether a false positive is occurring due to the nature of the correction equation (i.e. Cd 111 > 2 x LOQ, Cd 111 > xCd 111, and the RPD between Cd 111 and xCd 111 is > 20%). If so, evaluate whether reporting from the uncorrected Cadmium line (xCd 111) is appropriate. In this case, uncorrected Cd should only be reported when all of the following conditions are met:

    a. The corrected Cd line, "Cd 111", is greater than 2 x LOQ.

    b. The corrected Cd line, "Cd", is greater than the uncorrected Cd line, "xCd 111".

    c. The RPD between the corrected Cd line, "Cd 111", and the uncorrected Cd line, "xCd 111", is greater than 20%.

NOTE: When reporting from uncorrected Cd the ICSA standard may fail due to the presence of Mo. If this is the case, a non-conformance form and client approval is required.

14. There are some instances where significant interference is noted on both the primary and secondary isotopes for Se (masses 82 and 77). "xSe 78" is included in the methods for information only and is not a reportable isotope, however, in samples where both Se 82 and Se 77 read significantly higher than "xSe 78" a technical decision may be required regarding the Se analysis. Data from alternate methods (ICP-OES and/or GFAA) may need to be consulted, and if deemed appropriate the Se analysis may need to be switched to an alternate analytical method to achieve the most accurate determination.

C. When complete, check the following:

1. The beginning and end of the raw data are signed and dated by the reviewer and verifier.

2. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms.

3. Reread/redigest request forms are clipped to the front of the run.

4. The data are uploaded to Parallax via IDAT by the reviewer and verified from Parallax by the verifier. The person who uploads the run is responsible for ensuring that all data is uploaded using the correct units.

5. The raw data packet is placed in the verification bin. (For samples following Good Laboratory Practices [GLP], the raw data includes the "real-time" printout, as well as the final print file. The "real-time" printout should be signed and dated by the analyst).

D. Taking an instrument/analysis out of service/returning an instrument/analysis to service

NOTE: The following is taken from LOM-SOP-ES-222. In the event of an equipment failure, the following shall be performed:

1. Document the nature of the failure in the maintenance logbook

2. Document how and when the defect was discovered

3. Notification of supervisor or responsible person who can decide on appropriate action to take

4. The instrument must be clearly tagged as *Out of Service*. The tag must contain the following information:

   a. Date taken out of service

   b. Employee who took the instrument out of service

   c. Reason for tagout

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken to bring the equipment back into service.

7. Results of the corrective action (i.e., system calibration within specifications, etc.)

8. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument. This will be done on a case-by-case basis.

9. After repair, document whether the function has been fixed. Calibration or verification activities may need to be performed before the instrumentation is put back into service.

Table I

EPA 200.8
ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Tuning	Daily	No AMU diff. of >0.1 P.W. >=0.64 & <=0.66 %RSD < 5 for masses used for tuning	Perform full tuning for AMU. Adjust DAC values for P.W.
Daily Performance	Daily	Evaluated for information only	Instrument maintenance and optimization may be needed.
Calibration	The calibration will contain a blank and 1 standard.		
Initial Calibration Verification (ICV)	Must be analyzed immediately following calibration.	±10% of the true value	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be <LOQ.	Data for that analyte cannot be reported from the run (reanalyze).
Low Level Check (LLC)	Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB.	Statistical windows. Not applicable if sample concentrations are >10× the true value of the LLC.	Data for that analyte cannot be reported from the sample (reanalyze).
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA must be analyzed at the beginning and end of each run immediately following the LLC. The ICSAB must be analyzed at the beginning and end of each run immediately following the ICSA.	±20% of the true value for the analytes that are spiked. |ICSA| or |ICSAB| must be <2× LOQ for analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples	±15% of the true value.	Data bracketing the CCV for the affected analyte cannot be reported (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples	|CCB| must be <LOQ	Data bracketing the CCB for the affected analyte cannot be reported (reanalyze).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	|PB| must be <LOQ or 2.2× MDL whichever is greater. Samples >10× blank value can be taken.	Redigest all associated samples.
Laboratory Fortified Blank (LCS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	±15% of the true value	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data may be used.

Table I - Continued

EPA 200.8
ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Laboratory Fortified Blank Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	±15% of the true value	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data may be used.
Matrix Spike (MS)	Rate of 10% of analytical samples	Use statistical limits or the method limit of ±30% whichever is tighter.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4x the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less	RPD must be <20%	'*'-Flag the data package forms.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	±15%	Report % rec. on FormVB-IN.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50 MDL.	The percent difference must be <10%	'E'- Flag data on DP Forms
Samples		Sample reading must be within the linear range (see Definition 23).	Reanalyze at dilution that will bring sample concentration within the linear range
Linear Range Standard (LRS)	LRS not evaluated on all runs and may be used as needed.	±10% of the true value	Samples reading greater than the calibration range must be reanalyzed.
Internal Standards	Added to all samples by way of second pump channel.	Must be 60%-125% of the Calibration Blank	Reanalyze @ DF2

Table II

EPA-6020 Revision 0 Sept. 1994
ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Tuning	Daily	No AMU diff. of >0.1 P.W. >=0.64 & <=0.66 %RSD < 5 for masses used for tuning	Perform full tuning for AMU. Adjust DAC values for P.W.
Daily Performance	Daily	Evaluated for information only	Instrument maintenance and optimization may be needed.
Calibration	The calibration will contain a blank and 1 standard.		
Initial Calibration Verification (ICV)	Must be analyzed immediately following calibration.	±10% of the true value	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be <3*IDL. Not applicable if sample concentrations are >10× the value of |ICB|.	Data for that analyte cannot be reported from the run (reanalyze).
Low Level Check (LLC)	Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB	±50% of the true value Not applicable if sample concentrations are >10× the true value of the LLC	Data for that analyte cannot be reported from the sample (reanalyze).
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA must be analyzed at the beginning of each run or every 12 hours, which ever is more frequent.	±20% of the true value for analytes that are spiked. |ICSA| or |ICSAB| must be <2× LOQ for analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples	±10% of the true value.	Data bracketing the CCV for the affected analyte cannot be reported (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples	|CCB| must be <3*IDL. Not applicable if sample concentrations are >10× the value of |CCB|	Data bracketing the CCB for the affected analyte cannot be reported (reanalyze).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	|PB| must be < LOQ Not applicable if analyte reading in the sample is >20× the PB reading or <LOQ	Redigest all associated samples.
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, whichever is tighter for water LCSs. Use statistical limits for soil LCSs.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data may be used.

Table II - Continued

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ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, whichever is tighter for water LCSs. Use statistical limits for soil LCSs.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data may be used.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±25% whichever is tighter. RPD must be <20%	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4x the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less	If the samples are >100*IDL the RPD must be <20.	Flagged in data package and in the QC Summary.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	±15% of the true value	The data is flagged in the data package.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >100x MDL.	The percent difference must be <10%.	The data is flagged in the data package.
Samples		Sample reading must be within the linear range (see Definition 23).	Reanalyze at dilution that will bring sample concentration within the linear range
Linear Range Standard (LRS)	LRS not evaluated on all runs and may be used as needed.	±10% of the true value	Samples reading greater than the calibration range must be reanalyzed.
Internal Standards	Added to everything through use of second pump channel	30%-120% for samples 80%-120% for ICV, CCVs, ICB, & CCBs 30%-120% for LLC,ICSA, & ICSAB	Reanalyze @ DF5. Terminate analysis & reanalyze. Reanalyze.

Table III

CLP 5.2
ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Tuning	Daily	No AMU diff. of >0.1 P.W. <0.65	Perform full tuning for AMU. Adjust DAC values for P.W.
Daily Performance	Daily	%RSD <5. (see Manufacturer's chart)	Terminate, investigate, and repeat Daily Performance.
Calibration	The calibration will contain a blank and 1 standards		
Initial Calibration Verification (ICV)	Must be analyzed immediately following calibration.	±10% of the true value	Data for that analyte cannot be reported from the run (reanalyze).
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV.	|ICB| must be < CRQL.	Data for that analyte cannot be reported from the run (reanalyze).
Low Level Check (CRI)	Must be analyzed at the beginning and end of each run and before the ICSA and ICSAB, and at a minimum of once per 20 analytical samples.	±30% of the true value (±50% for Co, Mn, and Zn).	Data for that analyte cannot be reported from the sample (reanalyze).
Interference Check Standard A and AB (ICSA/ICSAB)	The ICSA/AB must be analyzed at the beginning/end of each run and every 20 samples (the CRI, ICSA, and ICSAB count as samples).	±20% of the true value for the analytes are spiked. |ICSA| or |ICSAB| must be <3× CRQL for the analytes that are not spiked.	Data for that analyte cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed immediately following the ICSAB and at a frequency of every 10 samples, and immediately after last sample.	±10% of the true value.	Data bracketing the CCV for the affected analyte cannot be reported (reanalyze).
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCV's at a frequency of every 10 samples	|CCB| must be <CRQL.	Data bracketing the CCB for the affected analyte cannot be reported (reanalyze).
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	|PB| must be <CRQL. Samples >10× blank value and samples <CRQL can be taken.	Redigest all associated samples.
Laboratory Control Standard (LCS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	±20% of the true value.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data may be used.

Table III - Continued

CLP 5.2
ICP-MS Metals

	Frequency	Acceptance	Corrective Action
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	±20% of the true value.	Redigest all associated samples. If the LCS or LCSD is OOS high and the sample reads < than its reporting limit, the data may be used.
Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	±25% of the true value.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4× the spike added a PDS must be performed.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less	If the samples are >5*CRQL the RPD must be <20.	'*'-Flag the DP Forms.
Post Digestion Spike (PDS)	Must be prepared with each background sample. Evaluated when matrix spike is not within specification.	±15% of true value.	Report % rec. on FormVB-IN.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50 MDL.	The percent difference must be <10%.	'E'- Flag data on DP Forms
Samples		Sample reading must be within the linear range (see Definition 23).	Reanalyze at dilution that will bring sample concentration within the linear range, but not less than ½ of the calibration range.
Linear Range Standard (LRS)	LRS not evaluated on all runs and may be used as needed.	±10% of the true value	Samples reading greater than the calibration range must be reanalyzed.
Internal Standards	Added to all samples by way of second pump channel.	60%125% of the calibration blank reading	Reanalyze @ DF2

APPENDIX C

Standard Operating Procedure
Gravel Sample Collection and Processing

Sample Collection and Handling of Cap Material at the Honeywell Celotex Site

Introduction

This Standard Operating Procedure (SOP) describes the procedures for sample collection, field and laboratory processing, and analysis of the cap material at the Honeywell Celotex Main Site. Deviations from this SOP may be required to collect a representative sample due to the nature of the materials being collected and site conditions. The Field Team Leader (FTL) will discuss any proposed changes with the project chemist and document any deviations from this SOP in the field logs and will describe those deviations in the Main Site Evaluation Report.

Sample Collection

The gravel cap, soil fill, and clay cover materials at each boring location will be penetrated with a hollow stem auger (HSA) to the approximate depth of surrounding grade, to the depth which constitutes the bottom of the 2-foot clay cover material, or until Main Site demolition debris is encountered. Based on currently available information this depth could range from at least 2 feet below ground surface (bgs) up to 6 feet bgs. Cap, cover, and fill materials will be logged and collected continuously with a split spoon, slit barrel or equivalent sampler (depending upon site conditions) with sufficient diameter so that the gravel fraction of the cap material will not prevent sample recovery. Once the entire cap thickness is penetrated, a conventional sized soil sampling device will be used to collect samples of the cover and fill materials for physical characterization, in order to reduce the amount of soil cuttings generated by the sampling activity. Once the limits of the boring are reached based on the identified criteria, augering and soil sampling will cease. Further details regarding the field work is identified in the Main Site Evaluation Work Plan (CH2M HILL, August 2006).

Sample Handling

Prior to collecting samples, the FTL will need to estimate the weight of the fine materials present in the gravel cap. A minimum of 200-300 grams of fine material (see below for estimating fines) will be required by the laboratory to perform the required analyses. Wherever a field duplicate is required, two times the sample volume will be collected at the location to obtain 400-600 grams of fine materials. In addition, at locations where a matrix spike/matrix spike duplicate (MS/MSD) is required, three times the sample volume will be collected to obtain 600-900 grams of fine materials.

A procedure similar to the following should be used to estimate the weight of the fine material present in the cap material.

1) At a location representative of the majority of sampling locations, collect a sample using a split spoon sampler or equivalent and weigh the entire sample without excluding any material. Record the weight as "Sample Gross Weight".

2) Use a screen/sieve to remove the 2 centimeter and larger particles. Record the weight after removal of > 2 cm particles as "Sample Total Weight".

3) Use an 8a 10-mesh screen/sieve to remove the particles remaining greater than 2.4 millimeters. Record the weight after removal of > 2.4 millimeters as "Sample Fines Weight".

4) Subtract 15 grams from Sample Fines Weight to account for the VOC sample that will be collected at each location and then multiply by 4 (the number of discrete samples to be composited). This theoretical number represents "Fines per Composite Weight"

5) If the approximate mass of Fines per Composite is greater than 200 grams, assume that the sampling approach will meet the objectives, if not, more sample is required from each discrete sampling location to achieve the 200 gram minimum after composite.

When collecting the discrete samples of the cap material to be composited prior to submission to the laboratory for analysis, the field team will process the samples in the following manner.

1) Cap material size fractions greater than 2 centimeters in diameter will be manually discarded in the field. A screen/sieve will be used to perform the size exclusion and the excluded material will be left in place at the sampling site.

2) The gravel-sized fraction (less than 2 centimeters and greater than 2.4 millimeters in diameter) and fines (less than 2.4 millimeters in diameter) of the cap material composite sample will both be submitted to the laboratory for sieving and analysis.

3) VOC samples will be collected using a sampling device such as an Easy Draw Syringe or equivalent and placed into two 40 mL VOC vials filled with 5 mL deionized water, and one 40 mL VOC vial filled with 5 mL methanol prepared by the laboratory. Upon return to laboratory, the exact mass of material in the vials will be determined. Samples collected with water will be frozen upon receipt at the laboratory according to method SW846-5035A. The field team will not add any additional labels to the jar. The volume of sample needed to provide 5 grams of material per vial will be estimated in the field based on the density of the fines. Ideally, the weight of each vial (to the nearest 1 gram) will be recorded by the field team prior to shipment and a line drawn on the vial to show the approximate level of the soil/deionized water or methanol in the vial prior to shipment. At a minimum, the volume of the soil/deionized water or methanol mixture will be noted by drawing the approximate level of material in the vial.

4) Semi- and non-volatile analyses will be collected in glass jars. The volume requirements are specified in the Quality Assurance Project Plan (QAPP) addendum. Please note that the approximate weight of sample needs to be as calculated in the procedure above. The Fines to Total ratio needs to be accounted for.

5) Decontamination of the screen/sieves will be performed as outlined for the other sampling equipment in the Work Plan between locations.

6) The QAPP/QAPP addendum will be followed for chain-of-custody completion, sample preservation, documentation requirements, sample packaging and shipping.

Laboratory Sample Handling and Analysis

The laboratory will receive approximately 73 composite samples of the cap material for analysis, including 7 duplicates and 4 MS/MSD pairs The following procedures will be used to prepare the samples for analysis.

1) The laboratory will weigh and record the weight of each sample received prior to sieving the sample with an 8a 10-mesh screen/sieve.

2) The weight of the fines collected after sieving will be recorded. The fines will be stored in glass jars at four degrees Celsius prior to analysis.

3) The screen/sieve will be decontaminated between each use following the laboratory's SOP for decontamination of sampling equipment/laboratory supplies. At a minimum, the following decontamination procedures will be followed:

    a) Lightly brush the screen/sieve to remove larger particles.

    b) Wash the screen/sieve in hot water with a light detergent soap.

    c) Rinse the screen/sieve with deionized water

    c) Dry the screen/sieve

The above steps will be performed for each sample. Initially (and possibly periodically), the laboratory will collect a rinseate blank after the decontamination process to determine the impact of the screen/sieve on the data and include those data in the report. In the event that the rinseate data indicate the screening/sieving are having a deleterious impact to the sample data, the laboratory will contact the CH2M HILL Project Chemist/Project Manager as soon as possible.

Analysis will be performed on the fines collected per the QAPP and standard laboratory procedures. The analyses that will be performed on the samples are as follows: SVOCs by SW846-8270C, Pesticides by SW846-8081A, Herbicides by SW846-8151A, Aroclors by SW846-8082, moisture content, and Total/SPLP metals (As, Be, Cd, Cr, Pb, Hg, Ni, Cu, Se, Ag, Tl and Zn) by SW846-6000/7000 series methods. VOC analyses, by Method 8260, will be performed on the discrete samples provided and not from the process described above.

The laboratory will adhere to the holding-times annotated in the QAPP addendum for preparation and analysis.

Laboratory Data Reporting

The data collected from the analysis of the fine materials will be reported by the laboratory based on the basis of the fines produced during the laboratory sieving operation. The

masses of the gravel size fraction (>2.4 mm and <2 cm) and the mass of fines (<2.4 mm) used for the VOC analysis can be used to adjust the concentrations reported by the laboratory to a concentration based on the "total sample" corrected for moisture content. The "total sample" includes the mass of the sample for the gravel sized fraction (>2.4 mm and <2 cm) and the sample mass from the fines (<2.4 mm). The masses of the gravel size fraction and fines are corrected for moisture content. The "total sample" includes the mass of the sample for the gravel sized fraction (> 2 mm and < 2 cm) and the sample mass from the fines (< 2 mm). If the laboratory's data system is not capable of reporting the data using the aforementioned approach, the data from the fines will be reported on a dry weight basis and the information below will be included with the analytical report:

1) The weight of the total sample submitted and the weight of the fine material created during the sieving will be provided.

2) The laboratory case narrative will detail the sample handling procedures that the laboratory followed, referencing this SOP.

3) The case narrative will annotate that the sample results are representative of the fines produced by sieving the sample and not the "total sample".

CH2M HILL Data Reporting

CH2M HILL will perform the necessary calculations and use the calculated results for data evaluation. CH2M HILL will also update the electronic data deliverable (EDD) with the "total sample" results prior to loading to the project database (LOCUS Focus).

The following steps will be used to determine the concentration of the "total sample":

1) Determine the ratio of the fine material to the "total sample". For example, one kilogram of "total sample" is determined in step 2 of the section above titled "Laboratory Sample Handling and Analysis" and 200 grams of fine material are determined in step 3 of the same section. The ratio of fines to the total material is 1:5.

2) The "total sample" result will be calculated by taking the results of the fine material (already adjusted for moisture content) and multiplying the data by the ratio. For example, copper is found at 10 mg/Kg in the fine material and the ratio of fines/"total sample" is 1:5. The "total sample" result is 10 mg/Kg multiplied by 0.2 for a result of 0.2 mg/Kg. The 0.2 mg/Kg result would be used for project decision making.